A1. TITLE AND APPROVAL SHEET

REMEDIAL INVESTIGATION FOR OPERABLE UNIT 3 LIBBY ASBESTOS SUPERFUND SITE

QUALITY ASSURANCE PROJECT PLAN

ESTIMATION OF ASBESTOS LEVELS IN SMOKE USING A BURN CHAMBER

Revision 0 – September 13, 2011

Prepared by
U.S. Environmental Protection Agency
Region 8
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LIST OF ACRONYMS

AOC	Administrative Order on Consent
CAR	Corrective Action Request
CEM	Continuous Emissions Monitor
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COC	Chain-of-Custody
DAS	Data Acquisition System
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EPA	U.S. Environmental Protection Agency
FS	Feasibility Study
FSDS	Field Sample Data Sheets
FTP	File Transfer Protocol
GO	Grid Opening
GPS	Global Positioning System
ID	Identification number
IL	Inter-laboratory
ISO	International Organization for Standardization
IUR	Inhalation Unit Risk
KDC	Kootenai Development Corporation
LA	Libby Amphibole
MCE	Mixed Cellulose Ester
MDEQ	Montana Department of Environmental Quality
MWH	MWH Americas, Inc
NDIR	Nondispersive Infrared Sensor
NMOC	Non-methane Organic Compounds
NVLAP	National Voluntary Laboratory Accreditation Program
OBTF	Open Burn Test Facility
OU	Operable Unit
PCM	Phase Contrast Microscopy
PCME	Phase Contrast Microscopy Equivalent
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RBC	Risk-Based Concentration
RD	Recount Different
RI	Remedial Investigation
RPM	Remedial Project Manager
RS	Recount Same

RTP	Research Triangle Park
SOP	Standard Operating Procedure
STEL	Short Term Exposure Limit
TEM	Transmission Electron Microscopy
THC	Total Hydrocarbons
TWA	Time Weighted Average
TWF	Time-Weighting Factor
USFS	U.S. Forest Service

A3. DISTRIBUTION LIST

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Copies of the QAPP will be distributed to the individuals above by EPA's contractor (SRC, Inc.), either in hard copy or in electronic format (as indicated). SRC will distribute updated copies each time a QAPP revision occurs.

A4. PROJECT TASK ORGANIZATION

Project Management

The U.S. Environmental Protection Agency (EPA) is the lead regulatory agency for Superfund activities within operable unit 3 (OU3). The EPA Remedial Project Manager (RPM) for OU3 is Christina Progess, EPA Region 8. Ms. Progess is a principal data user and decision-maker for Superfund activities within OU3.

The Montana Department of Environmental Quality (MDEQ) is the support regulatory agency for Superfund activities within OU3. The MDEQ Project Manager for OU3 is John Podolinsky. EPA will consult with MDEQ as provided for by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan, and applicable guidance in conducting Superfund activities within OU3.

EPA has entered into an Administrative Order on Consent (AOC) with Respondents W.R. Grace & Co.-Conn. and Kootenai Development Corporation (KDC) for performance of a Remedial Investigation/Feasibility Study (RI/FS) at OU3 of the Libby Asbestos Site. Under the terms of the AOC, W.R. Grace & Co.-Conn. and KDC will implement the activities described in this document, under EPA supervision. The designated Project Coordinator for Respondents W.R. Grace & Co.-Conn. and KDC is Robert Medler of Remedium Group, Inc. He is assisted by Robert Marriam of Remedium Group, Inc.

Technical Support

Ms. Progess will receive technical support on this project from a number of EPA staff and contractors, including:

- Dr. Robert Edgar is the EPA Region 8 technical lead for this project, and will help ensure project objectives and study design are well planned.
- Dr. Dan Wall is EPA's lead ecological risk assessor for the OU3 site, and will help ensure the data are helpful for risk assessment purposes.
- Dr. William Brattin of SRC, Inc. will assist in the development of quality assurance project plans (QAPPs) and standard operating procedures (SOPs), and will assist in data reduction and interpretation activities.
- Ms. Lynn Woodbury of CDM, Inc. will also assist in the development of QAPPs and SOPs, and will provide data management for the project.

Field Sampling Activities

All duff collection activities described in this QAPP will be performed by W.R. Grace & Co.-Conn. and KDC, in strict accordance with the sampling plans developed by EPA. W.R. Grace & Co.-Conn. and KDC will be supported in this field work by MWH Americas, Inc. (MWH). The project manager for MWH is John Garr.

Burn Chamber Studies and Measurements

Burn chamber studies will be conducted at EPA's research facility in Research Triangle Park (RTP), North Carolina, under the supervision and direction of Dr. Paul Lemieux. Dr. Lemieux will be responsible for implementing the studies and collecting the data measurements described in this QAPP.

Asbestos Analysis

All samples of asbestos collected as part of this project will be sent for preparation and/or analysis at laboratories selected and approved by EPA. Laboratories that will be utilized for analysis asbestos samples may include Hygeia Laboratories, Inc. and/or EMSL Analytical, Inc.

Data Management

Administration of the master database for OU3 will be performed by EPA contractors. The primary database administrator will be Lynn Woodbury of CDM. She will be responsible for sample tracking, uploading new data, performing data verification and error checks to identify incorrect, inconsistent or missing data, and ensuring that all questionable data are checked and corrected as needed. When the OU3 database has been populated, checked and validated, relevant asbestos data may be transferred into a Libby Asbestos Site database, as directed by EPA for final storage.

EPA Quality Assurance Officers

The EPA Quality Assurance Officer for this project is Rebecca Thomas. Ms. Thomas is independent of the entities planning and obtaining the data, and is responsible for ensuring that this QAPP is prepared in accordance with EPA guidelines and requirements. The EPA Quality Assurance Officer for the Burn Chamber studies is Ms. Eletha Brady-Roberts of EPA's National Homeland Security Research Center.

Organizational Chart

Figure A-1 presents an organizational chart that shows lines of authority and reporting responsibilities.

A5. PROBLEM DEFINITION/BACKGROUND

A5.1 Site Background

Libby is a community in northwestern Montana that is located near a large open-pit vermiculite mine. Vermiculite from the mine at Libby is known to be contaminated with amphibole asbestos that includes several different mineralogical classifications, including mainly richterite and winchite with lower levels of tremolite and possibly actinolite. For the purposes of EPA investigations at the Libby Asbestos Superfund Site, this mixture is referred to as Libby Amphibole (LA).

Historic mining, milling, and processing of vermiculite at the site are known to have caused releases of vermiculite and LA to the environment. Inhalation of LA associated with the vermiculite is known to have caused a range of adverse health effects in exposed humans, including workers at the mine and processing facilities (Amandus and Wheeler 1987, McDonald et al. 1986, McDonald et al. 2004, Sullivan 2007, Rohs et al. 2007), as well as residents of Libby (Peipins et al. 2003). Based on these adverse effects, EPA listed the Libby Asbestos Superfund Site on the National Priorities List in October 2002.

Starting in 2000, EPA began taking a range of cleanup actions at the site to eliminate sources of LA exposure to area residents and workers using CERCLA (or Superfund) authority. Given the size and complexity of the Libby Asbestos Site, EPA designated a number of OUs. OU3 includes the property in and around the former vermiculite mine and the geographic area surrounding the mine that has been impacted by releases and subsequent migration of hazardous substances and/or pollutants or contaminants from the mine.

EPA is concerned with environmental contamination in OU3 because the area is used by humans for logging, a variety of recreational activities, and, in the case of U.S. Forest Service (USFS) employees, land management and fire fighting activities. The area is also habitat for a wide range of ecological receptors (both aquatic and terrestrial). Contaminants of potential concern to EPA in OU3 include not only LA, but any other mining-related contaminants that may have been released to the environment.

A5.2 Reasons for this Project

Historic releases of LA to the environment in OU3 have resulted in contamination of soil, tree bark, and duff (organic litter and debris on the forest floor) in the area surrounding the mine. When wildfires occur in OU3, it is expected that fibers in duff and bark may be released into the air. This could result in inhalation exposures to USFS workers fighting the fires, either on the ground or in the air, and (depending on wind direction and meteorological conditions) might also result in exposure of residents of Libby. However, available data are not adequate to support reliable quantitative estimation of the air concentrations of asbestos fibers that may occur in smoke during a wildfire in OU3.

In order to obtain information needed to evaluate exposures to LA in smoke from fires in OU3, EPA will collect data using two basic strategies:

- Opportunistic Field Measurements. In this approach, when authentic wildfires occur in OU3, samples of air will be collected in the vicinity of USFS workers fighting the fires on the ground and in the air, at a location in the downwind smoke plume, and in the community of Libby.

- Combined Laboratory Studies/Modeling Approach. In this approach, measurements of LA in smoke (fibers per unit particulate matter in smoke) are collected under controlled conditions in a laboratory-scale simulation, and the measured values are combined with USFS models that predict smoke particulate matter levels during fires to yield predicted concentrations of LA in air.

This QAPP describes the plan for collecting measurements of LA fibers per unit particulate matter in smoke from burning contaminated source materials from OU3 for use in the combined laboratory study/modeling approach. The plan for collecting opportunistic measurements during authentic wildfires in OU3 is presented in a separate document (EPA 2011).

A5.3 Applicable Criteria and Action Limits

At present, there are no criteria or action limits that apply specifically to exposure of firefighters or other individuals to LA in smoke. More general criteria for exposure of workers to asbestos in workplace air have been established by the Occupational Safety and Health Administration (OSHA). The short-term (15 minute) exposure limit (STEL) is 1.0 fiber per cubic centimeter of air (f/cc), and the longer-term time-weighted average (TWA) exposure limit is 0.1 f/cc. Both exposure limits are expressed in terms of phase contrast microscopy (PCM) fibers.

A6. PROJECT/TASK DESCRIPTION

Task Summary

Basic tasks that are required to implement this QAPP include the following:

- Collect duff material that will be burned from an appropriate location in OU3
- Ship the material to RTP
- Implement burn chamber studies as described in this QAPP, collecting samples of smoke for analysis of LA and particulate matter
- Analyze samples of duff, smoke, and ash for LA and other indicators
- Calculate the concentration of LA in smoke per unit particulate matter released per unit concentration of LA in the burn material
- Model the concentration of LA in smoke that may be breathed by firefighters or other individuals exposed to smoke from fires in OU3
- Calculate the level of human health risk associated with exposure to smoke

Each of these basic tasks is described in greater detail in subsequent sections of this QAPP.

Work Schedule

The work schedule for performing these tasks begins with collection of the burn material to be used in these studies. This task requires that collection occur under dry conditions, so this task must be completed in middle to late summer of 2011.

Burn chamber studies, sample analysis, and data evaluation and interpretation tasks will be performed between fall of 2011 and spring of 2012. The goal is have results and conclusions available before the start of the wildfire season in late spring of 2012.

Locations to be Studied

The location where duff will be collected is described in Section B2.1.

Resources and Time Constrains

As noted above, the first time constraint is that duff must be collected from OU3 when conditions are dry and warm, and before rain and snow begin to occur in the fall. The second time constraint is to obtain the data and evaluate the results before the 2012 fire season begins. This is important in helping the USFS select the most appropriate strategy for fire fighting in OU3.

A7. QUALITY OBJECTIVES AND CRITERIA

Performance Criteria

The range of LA concentrations that will occur in burn chamber smoke is not known. However, it is possible to estimate the concentration levels that would correspond to a level of human health concern. These calculations are provided in Section B4. The analytical requirements for LA measurements established in Section B4 are such that concentrations of LA in burn chamber smoke will be reliably detected and quantified if present at levels of concern.

Precision

The precision of asbestos measurements is determined mainly by the number (N) of asbestos fibers counted in each sample. The coefficient of variation resulting from random Poisson counting error is equal to $1/N^{0.5}$. In general, when good precision is needed, it is desirable to count a minimum of 3-10 fibers per sample, with counts of 20-25 fibers per sample being optimal.

Bias and Representativeness

It is expected that LA levels in smoke may vary widely as a function of fire location, burn conditions, and meteorological conditions. Consequently, obtaining data that are fully representative of this wide range of potential levels of LA in smoke is difficult. The burn materials selected for use in this study are specifically intended to represent the high-end of what may occur in OU3, so absolute levels of LA in burn chamber smoke are likely to be biased high. However, the data reduction protocol normalizes for this by expressing results in terms of LA fibers released per unit concentration in duff. This approach should help ensure that results are not biased and are useful in predicting releases from fires at a range of representative locations in OU3.

Completeness

Target completeness for this project is 100%. If any samples of smoke are not collected, or if LA analysis is not completed successfully, this could result in that portion of the study providing no useful information.

Comparability

The data generated during this study will be obtained using standard analytical methods for LA and will yield data that are comparable to existing and future analyses of LA in air and smoke.

Method Sensitivity

The method sensitivity (analytical sensitivity) needed for LA in smoke and other media is discussed in Section B4.

A8. SPECIAL TRAINING/CERTIFICATIONS

Field Personnel

Asbestos is a hazardous substance that can increase the risk of cancer and serious non-cancer effects in people who are exposed by inhalation. All individuals involved in the collection, packaging and shipment of burn material from OU3 must have OSHA 40-hour health and safety training, and must wear appropriate personal protective equipment.

It is the responsibility of Remedium, Inc., or their contractors, to ensure that sampling is conducted in accordance with the project Health and Safety Plan (HASP) and to maintain appropriate documentation of training by active field personnel.

Laboratory Certification

All laboratories that analyze samples smoke, duff, or ash for asbestos as part of this project must participate in and have satisfied the certification requirements in the last two proficiency examinations from the National Institute of Standards and Technology/National Voluntary Laboratory Accreditation Program (NVLAP). Laboratories must also have demonstrated proficiency by successful analysis of Libby-specific performance evaluation samples and/or standard reference materials and must participate in the on-going laboratory quality assurance program for the Libby OU3 project.

It is the responsibility of EPA to ensure that these requirements are satisfied and to ensure that appropriate documentation of laboratory certification is available in laboratory files.

A9. DOCUMENTATION AND RECORDS

Field Documentation

The field sampling team will maintain a field log book. The log book shall be a record of all potentially relevant information on duff sampling activities and conditions. Examples of the type of information to be recorded in the field log include:

- Names of team members
- Current and previous weather conditions

- Field sketches
- Number and type of samples collected
- Any special circumstances that influenced sample collection

As necessary for sample collection and location documentation, photographs will be taken using a digital camera. Global positioning system (GPS) coordinates will be recorded for the approximate central point of all sampling areas.

Burn Facility Documentation

A detailed QAPP for all activities performed at the burn chamber facility is provided in Appendix A. This QAPP describes all documentations and records that will be generated at this facility during the project.

Analytical Laboratory Documentation

All analytical data for LA generated in the analytical laboratory will be documented on laboratory bench sheets. The data from these bench sheets will then be transferred into project-specific electronic data deliverable (EDD) spreadsheets, as provided in Appendix C.

B1. BURN CHAMBER STUDY DESIGN

Measurements of LA release from the burning of contaminated source material from OU3 will be performed at the EPA Open Burn Test Facility (OBTF) in RTP.

The burning will occur in a burn chamber placed in an enclosed shed (burn hut). Fuel is placed in the burn chamber, and is replenished during the burn by addition of new fuel through a feed chute. Air for the fire is provided by a fan that blows air into the burn hut. Smoke from the burning material travels through an exhaust flue, where sampling for LA and particulate matter is performed. A schematic diagram is shown in Figure B1.

Because so little is known about the levels of LA that may occur in smoke or the best way to collect reliable data, the study will be performed in phases. The first phase (Phase I) is intended to identify key variables that influence the release of LA from burning material, to provide initial estimates of LA concentration values in smoke, to provide initial estimates of the partitioning of LA fibers between air emissions and ash, and to gain experience on the best way to perform the burn and to collect reliable data.

Additional phases of investigation will be planned and implemented, as may be needed, after collection and evaluation of data from the Phase I studies.

B1.1 Burn Material

Type of Material

As noted above, the source materials most likely to release LA to air during a fire in OU3 are duff and bark. During an authentic wildfire, the principal material that is burned is duff and small woody debris, while bark on large standing trees (the likely primary location of embedded LA fibers) is usually only charred. In addition, available data collected during the Phase I investigation (EPA 2007) indicate that the levels of LA (mass per unit mass) are likely to be much higher in duff than in bark (see EPA 2010). Thus, it is considered likely that the main source of LA release to air during a fire will be duff. For this reason, the Phase I study will focus only on duff as the burn material.

Consequently, it is expected that data collected on the levels of LA released per unit mass of material burned will likely be higher for duff than for authentic fires in which other types of fuel (e.g., wood in fallen trees and branches, wood from young trees without LA contamination, etc.) are also burned. If additional studies are needed, burning fuels that contain various levels of wood along with the duff will be considered.

Amount of Material

The optimum burn rate for duff from OU3 is not yet known, but experience with chrysotile-containing building materials suggests that a burn rate of about 10 pounds per hour might be appropriate. Based on this, and assuming a burn duration of approximately one hour, the mass of duff required per burn is approximately 10 pounds. Assuming that a total of up to 6 different burns may be performed during Phase I (see Section B1.4), the total amount of duff required is a minimum of 60 pounds. To allow for study flexibility, approximately 90-120 pounds of duff material will be collected.

B1.2 Burn Chamber

The burn chamber will be constructed of a 6-inch tall segment of a cut 55-gallon drum with a metal mesh screen at the bottom suspended over a propane-fired circular burner of the type used for deep fryers such as those used for deep frying turkeys. The burner will be placed in a high walled pan or tray to collect ash.

The propane-fired burner will be capable of operating at a range of propane burn rates so that different temperatures and duff burn rates can be achieved. This is because the release rates of LA and/or particulate matter less than 2.5 um ($PM_{2.5}$) may depend on temperature. This will be investigated by performing initial burns both at a relative low temperature and at the high-end of what can be achieved with the burner.

The burn chamber will be fitted with three K-Type thermocouples inserted in a radial fashion into the interior of the burn chamber to measure temperatures of the burning mass of material. Two additional thermocouples will be positioned above the burn chamber to measure the temperature above the flame zone.

The burn chamber will be mounted on a scale with a resolution of about ± 0.1 pound to continuously monitor the mass of the fuel remaining. The propane tank is not mounted on this scale, so the loss of propane during the burn will not confound the mass measurements.

B1.3 Burn Protocol

Burns will be initiated by starting the propane burner and stabilizing its operation for 5 minutes. Duff combustion will be initiated by feeding three bags (~1.5 pounds) of duff into the burn chamber. For convenience and also for safety, the duff will be added as packaged in paper bags, and not removed from the bags. The mass of paper in the bags is small compared to the mass of duff, and is not expected to substantially alter the release rate of $PM_{2.5}$.

The mass of duff in the burn chamber will be continuously monitored during the burn using the scale in the burn chamber, either visually and/or by weight using the scale attached to the burn unit. Whenever the amount of duff remaining has decreased by about 1/3 to 1/2 (e.g., from about 1.5 pounds to about 1 pound), a new bag of duff shall be added *via* the feed chute to maintain an approximately constant amount of burn material. Each burn shall be carried out for a period of about one hour.

B1.4 Study Variables

It is anticipated that the temperature at which the material is burning may influence the release of both LA and particulate matter. For this reason, the pilot study will investigate releases at a relatively low and a relatively high burn temperature. Three burns will be conducted at each temperature to provide data on statistical variability between replicate experiments.

The temperature of the burn will be controlled by either adjusting the flame of the propane burner and/or the rate of air flow into the burn hut. Target temperatures are approximately 800 (low temperature) and 1600 (high temperature) degrees Fahrenheit (ºF). It is understood that this range may not fully encompass the temperatures that may occur during authentic wildfires, but this range is likely to be adequate to determine how sensitive the results (both LA and $PM_{2.5}$ release) are to burn temperatures.

B1.5 Critical Measurements

The critical measurements associated with each burn study are the amounts (concentrations) of LA and PM2.5 released into smoke, along with the concentration of LA in the burn material. Other measurements that characterize the burn conditions (e.g., temperature, burn rate) and characteristics of the smoke are less vital, but are important to help understand what factors influence the release of LA into smoke.

B1.6 Data Reduction and Interpretation

Data generated from burn chamber studies will be utilized as follows:

Step 1: Calculate the emission factor of LA from the combustion of duff as follows:

$$EF_{duff}(LA) = \frac{C_{flue}(LA) \cdot Q_{total}}{m_{duff} \cdot C_{duff}(LA)}$$

where:

$EF_{duff}(LA)$ = emission factor of LA from duff combustion (structures emitted per structure in material burned)

$C_{flue}(LA)$ = concentration of LA in flue duct effluent (s/m^3)
$C_{duff}(LA)$ = concentration of LA in the duff, dry basis (s/kg duff)
Q_{total} = total duct gas flow rate (m^3/hr)
m_{duff} = the mass feed rate of duff in units of mass per unit time (kg duff/hr)

Step 2: Calculate the emission factor of $PM_{2.5}$ from the combustion of duff as follows:

$$EF_{PM\,2.5} = \frac{C_{PM\,2.5} Q_{total}}{m_{duff}}$$

where:

$EF_{PM2.5}$ = the emission factor of $PM_{2.5}$ from duff combustion (mg emitted per kg duff burned)
$C_{PM2.5}$ = the concentration of $PM_{2.5}$ in flue duct effluent, dry basis (mg/m^3)

Alternately, $EF_{PM2.5}$ can be extracted from available published sources, such as the AP-42 Emission Factor database[1].

Step 3: Calculate the ratio of LA to $PM_{2.5}$ in flue gas per unit concentration in duff as follows:

$$R = \frac{EF_{duff}}{EF_{PM\,2.5}}$$

where:

R = LA fibers emitted per mg $PM_{2.5}$ emitted per s/kg in the duff that is burned

By using this approach, the emission rate of LA can be calculated over a range of values of $C_{duff}(LA)$ representing the LA concentration in the duff at different spatial locations in OU3.

Step 4: Use the USFS Smoke Impact Spreadsheet (SIS) model[2] to predict the concentration of $PM_{2.5}$ in air (mg/m^3) at an exposure point of interest near a fire in OU3.

Step 5: Estimate the concentration of LA in air at the exposure point as follows:

$$C_{air}\,(\text{LA f/cc}) = R \cdot C_{duff} \cdot PM_{2.5} \cdot 1E\text{-}06$$

where:

R = ratio (LA fiber/cc per mg/m^3 $PM_{2.5}$ per fiber/kg in duff)
C_{duff} = concentration of LA in duff (LA fibers/kg duff)

[1] http://www.epa.gov/otaq/ap42.htm
[2] http://www.airsci.com/SISmodel/SIS_Users_Manual-6.17.03.pdf

$PM_{2.5}$ = concentration of $PM_{2.5}$ in breathing zone air (mg/m^3)
1E-06 = unit conversion factor (m^3/cc)

Step 6: Estimate human health risk using the approach recommended by EPA (2008):

$$Risk = C_{air} \cdot TWF \cdot IUR_{a,d}$$

where:

Risk = Lifetime excess risk of developing cancer (lung cancer or mesothelioma) as a consequence of site-related asbestos exposure.

C_{air} = Concentration of LA in air (PCM or PCM-equivalent [PCME] f/cc)

TWF = Time-weighting factor; the value of the TWF term ranges from zero to one, and describes the average fraction of a lifetime during which exposure occurs from the specific activity being assessed.

$IUR_{a,d}$ = Inhalation unit risk (PCM $f/cc)^{-1}$ based on continuous exposure beginning at age "a" and continuing for duration "d" years

B2. SAMPLING METHODS

B2.1 Duff Collection

Sampling Location

Available data indicate that the concentration of LA in duff tends to be highly variable over space, although there is a general tendency for high values to occur near the mine in the downwind (northeast) direction (see Figure B2). Duff for Phase I will be collected from an area that is likely to be relatively high in LA, as indicated by the red triangle in Figure B2. This is the same general area where a study on exposure of small mammals to LA in duff was performed.

Because of the potential for small scale variability, to the extent that it is practical, the duff sample (90-120 pounds) should be a composite collected over a large area within the red triangle. This minimizes the probability of collecting duff from a sub-location where LA concentrations are low. Collecting and testing duff from other locations in OU3 with differing concentrations of LA may be performed as part of subsequent studies, as may be appropriate.

Duff shall be collected only during dry field conditions (i.e., no rain event > $1/10^{th}$ of an inch within the past 1 week, and no measurable rain within the past 48 hours).

Duff should be collected by hand, picking up all loose vegetative cover, including small sticks, pine cones, pieces of bark, etc. Care should be taken, to the degree possible, to exclude collection of mineral soil from below the duff layer. Sticks and other pieces of debris longer than about 4 inches shall be cut using garden shears into pieces of 2-3 inches in length. This is needed so that the material can be mixed and divided into bags, as described below.

Because duff collection and other handling will involve disturbing potentially contaminated material, collection of duff should be done only by trained and hazmat certified personnel outfitted with Level C (modified) personal protection equipment, including full Tyvek suits, certified-fitted full-face respirators fitted with high-efficiency particulate air (HEPA) filters, latex booties, and double gloves.

If difficulties or problems arise during the duff collection effort, the field team leader should immediately contact EPA (Christina Progess, Robert Edgar, and/or Dan Wall) to identify the problem and to recommend and discuss a remedy. The field collection protocol shall not be significantly modified without EPA approval. If changes are needed, EPA will prepare a field sampling modification that will be attached to the QAPP for documentation purposes.

B2.2 Burn Chamber Smoke Sampling

LA in burn chamber smoke will be sampled using two different techniques.

MCE Filter Method

The first method will be to draw air from the exhaust flue and pass it through a mixed cellulose ester (MCE) filter (25 mm diameter, 0.8 um pore size). The sampling port for the MCE filters will be at a location in the exhaust flue where gas temperatures are sufficiently low that neither the filter nor the filter cassette is damaged by high temperature.

The optimum flow rate and duration for collection of MCE filters are not yet known, so initial conditions will utilize a flow rate of 5 liters/min, and a sampling time of 15 minutes per sample. This will result in 4 MCE filters per 1-hour burn, with a volume of 75 liters per filter. These parameters may be revised during Phase I as experience is gained and data are collected.

The flow rate through the filter will be monitored during sample collection. If the flow begins to decrease due to filter plugging with particulate matter before 15 minutes, the filter should be changed more frequently, such that a flow rate of approximately 5 liters/min is maintained during the entire hour.

Impinger method

The impinger sampling method is essentially EPA Method 5[3], with the only variation that no filter is included in the sample train. The sampling port for the impingers will be in close proximity to the sample port for the MCE filter.

The sampling train consists of the probe, followed by 4 impingers: two each containing 100 mL of deionized water, one empty, and the last one containing silica gel. The two impinger water samples, along with the probe rinse water, are combined and submitted to the laboratory for analysis by transmission electron microscopy (TEM), as specified in Section B.4.2.

B2.3 Ash Sampling

After the burn is completed, all the ash remaining from the combustion of the duff will be collected and well-mixed. The total mass of the ash will be weighted and an aliquot of 10-20 grams of ash will be placed into a glass bottle and shipped to the analytical laboratory for analysis of LA in the ash as described in Section B4.

B3. SAMPLE HANDLING AND CUSTODY

B3.1 Bulk Duff Sample Handling and Shipping

Duff collected as above will be placed into one or more clean steel 55-gallon drums, as may be required. Once sufficient duff has been collected, the steel drum(s) shall be sealed by placing a lid on each drum, and the duff shall be mixed by rolling the drum(s) back and forth on its side for approximately 15 minutes. In addition, each drum shall be inverted (turned end to end) once every 5 minutes (a total of 3 times) during the mixing. This is intended to help minimize variability between different sub-samples of the duff. This is important because the Phase I burns will seek to investigate study variables that influence release rates, and this would be confounded if different batches of duff contained substantially different levels of LA.

Sample Packaging

Once the duff has been mixed, it will be packaged by placing random sub-samples of 0.5 ± 0.1 pounds into paper bags. The actual weight of duff in each bag will be determined by weighing, and marked on the bag with an indelible marker. Each bag will then be sealed by folding over the top and closing with staples. The bags will then be placed into one or more large shipping containers (e.g., 55-gallon blue polyethylene plastic drums) for shipment to RTP. A quantity of pre-dried desiccant (e.g., 5 pounds of silica gel, Dry-Rite or equivalent) will be placed into the

[3] http://www.epa.gov/ttn/emc/promgate/m-05.pdf

bottom of each drum prior to adding the bags of duff, to help prevent mold growth in the duff and to help prevent moisture degrading the structural integrity of the bags during shipment.

Sample Shipping

Drums of duff will be shipped to the following address:

Paul Lemieux
EPA, Research Triangle Park
109 T. W. Alexander Drive (E343-06)
Research Triangle Park, NC 27709
(919) 541-0962

Four bags of duff, selected at random by the field team, will be shipped to the following address for analysis of LA content:

Robyn Denton
EMSL Analytical, Inc.
200 Route 130 North
Cinnaminson, NJ 08077
856-303-2556

B3.2 Identification of Samples for Asbestos Analysis

All samples collected for asbestos analysis will be labeled with unique sample identification (ID) number. For the purposes of this study, this sample ID will be a "self-reading" ID that is generated by RTP at the time of sample collection. The format of the sample ID will depend upon the type of sample that is collected, as described below:

Bags of Duff

Random bags of duff shall be labeled as follows: DUFF##
where ## designates a number from 01 to 04

Example: DUFF02

MCE Air Filters

MCE air filters shall be labeled as follows: MCE- EXP##–TIME##–PORT##

where MCE indicates the collection method, EXP## indicates the experiment number, TIME## indicates the time interval during the experiment the sample was collected, and PORT## indicates the port in the exhaust flue from which the sample was collected.

Example: MCE-02-15to30-04

Impinger Filters

Impinger filters shall be labeled as follows: IMP- EXP##–TIME##–PORT##
where IMP indicates the collection method, EXP## indicates the experiment number, TIME## indicates the time interval during the experiment the sample was collected, and PORT## indicates the port in the exhaust flue from which the sample was collected.

Example: IMP-03-0to15-05

Burn Ash

Samples of ash from each burn shall be labeled as follows: ASH – EXP##
where EXP## indicates the experiment number

Example: ASH-01

Sample IDs should be written on each sample container in permanent marker or using labels created with indelible ink by the Burn Chamber study leader or authorized delegate.

B3.3 Filter Handling and Shipping

Sample packaging and shipping of air filters for the analysis of LA will follow the requirements described in OU3 SOP No. 8 (Revision 2) (see Appendix B). EPA will contact Remedium prior to sample shipment to determine how the samples will be divided between the two analytical labs, and will arrange for shipment accordingly.

Filters and other samples for the analysis of LA will be shipped to either EMSL Analytical or Hygeia Laboratories using the following addresses:

Ron Mahoney
EMSL Analytical, Inc.
107 W. 4th St.
Libby, MT 59923
406-293-9066

Kyeong Corbin
Hygeia Laboratories
82 W. Sierra Madre Blvd
Sierra Madre, CA 91024-2434
626-355-4711

To determine which laboratory is to receive and analyze samples, contact Bob Marriam, Remedium Group Consultant, at 901-820-2023 or robert.r.marriam@grace.com.

B3.4 Chain of Custody

All shipments of duff, ash, and filters will be performed utilizing proper chain-of-custody (COC) procedures, as detailed in OU3 SOP No. 8 (Revision 2) (see Appendix B).

The purposes of the COC form are: a) to establish the documentation necessary to trace possession from the time of collection to final disposal, and b) to identify the type of analysis requested. All corrections to the COC record will be initialed and dated by the person making the corrections. Each COC form will include signatures of the appropriate individuals indicated on the form. The originals will accompany the samples to the laboratory and copies documenting each custody change will be recorded and kept on file. One copy of the COC form will be kept by field personnel.

All required paper work, including sample container labels, COC forms, custody seals and shipping forms will be fully completed in indelible ink (or printed from a computer) prior to shipping of the samples to the laboratory. All shipping will occur through overnight delivery.

All samples that may require special handling by laboratory personnel to prevent potential exposure to LA or other hazardous substances will be clearly labeled.

Chain-of-custody will be maintained until final disposition of the samples by the laboratory and acceptance of analytical results.

B3.5 Holding Times

There are no holding times for samples of asbestos.

B3.6 Archival and Final Disposition

All sample materials, including duff, ash, filters, and grids will be maintained in storage at the analytical laboratory unless otherwise directed by EPA. When authorized by EPA, the laboratory will be responsible for proper disposal of any remaining samples, sample containers, shipping containers, and packing materials in accordance with sound environmental practice, based on the sample analytical results. The laboratory will maintain proper records of waste disposal methods, and will have disposal company contracts on file for inspection.

B4. ANALYTICAL METHODS

B4.1 Analysis of LA in Duff

Each bag of duff (four total) will be analyzed as a separate sample.

Duff analysis requires ashing of the duff followed by resuspension of the residue and analysis by TEM for LA structures, as described in SOP DUFF-LIBBY-OU3 (Revision 0) (see Appendix B). Results shall be expressed as LA structures per gram (s/gram) and mass percent (grams of LA per 100 grams of duff), both on a wet-weight (as received) and a dry-weight basis.

B4.2 Analysis of LA in Smoke Samples

B4.2.1 LA on MCE Filters

Analytical Method

All MCE filters collected during test burns will be analyzed by TEM in basic accord with ISO 10312:1995(E), applying all relevant Libby-specific laboratory modifications, including LB-000016, LB-000019, LB-00028, LB-000030, LB-000066, and LB-000085 (see Appendix B).

Indirect preparation

Because it is expected that all filters will be overloaded with particulate matter, it is anticipated that all filters will require indirect preparation as described in SOP EPA-LIBBY-08 (Revision 1) (see Appendix B). This shall include a low-temperature plasma ashing step to remove any organic particulate matter arising from incomplete combustion of the duff. Initial suspension of the ashed residue shall be performed in 10 mL of 5 N HCl to facilitate dissolution of any mineral salts that may be present. Once salts are dissolved, the acid is diluted to 100 mL with filtered and deionized water. Because indirect preparation may tend to overestimate true concentrations of asbestos in air, any use of the data to estimate human exposure will include a discussion of the uncertainty and potential bias associated with the use of indirect preparations.

Because the concentration of LA in the smoke is not known, the analyst should prepare several different indirect filters using different volumes of the suspension of ashed residue (e.g., 30 mL, 10 mL, 3 mL, 1 mL), seeking to obtain loading on the secondary filter that is optimal for analysis (< 25% total particulate loading, < 30 LA structures per grid opening).

Counting Rules

In brief, all particles with fibrous morphology, an x-ray diffraction pattern consistent with amphibole asbestos, a energy dispersive spectrum consistent with LA, length ≥ 0.5 um, and aspect ratio ≥ 3:1 will be counted and recorded.

Target Sensitivity

The target sensitivity for analysis of MCE filters from filter samples of the exhaust flue is calculated in a series of steps, as follows:

$$RBC_{bz} = \frac{C_{flue}(LA)}{C_{flue}(PM2.5)} \cdot C_{smoke}(PM2.5)$$

where:

RBC_{bz} = risk based concentration of LA in breathing zone air (s/cc)
$C_{flue}(LA)$ = concentration of LA in flue duct air (s/cc)
$C_{flue}(PM_{2.5})$ = concentration of $PM_{2.5}$ in flue duct air (ug/m^3)
$C_{smoke}(PM_{2.5})$ = concentration of $PM_{2.5}$ in breathing zone air near a fire (ug/m^3)

Rearranging this equation yields:

$$RBC_{flue}(LA) = RBC_{bz} \cdot \frac{C_{flue}(PM2.5)}{C_{smoke}(PM2.5)}$$

Values for these parameters are as follows:

RBC_{bz} = 0.0073 s/cc (see Attachment A)
$C_{flue}(PM_{2.5})$ = 80,000 ug/m^3 (estimated based on previous experience with burn chamber experiments)
$C_{smoke}(PM_{2.5})$ = 100 ug/m^3 (assumed value for smoke in breathing zone air near fires)

Based on these values, the risk-based concentration in flue duct air is approximately 5.9 s/cc. The target sensitivity is calculated as follows:

Target S_{flue} = RBC_{flue} / target count

For the purposes of planning the analytical requirements for Phase I of this study, the target count is set to 5 (i.e., when analyzing a sample whose true concentration is equal to the RBC, the number of particles that will be observed and counted will average about 5).

$$Target\ S_{flue} = \frac{5.9\ s/cc}{5\ s} = 1.2\ cc^{-1} \quad (rounded\ to\ 1\ cc^{-1})$$

Stopping Rules

Based on this, counting shall be performed as follows:

1) Examine at least two grid openings on each of at least 2 grids.
2) Continue examining grid openings until one of the following is achieved:
   - The target sensitivity is achieved
   - A total of 50 LA structures are counted
   - A total of 100 grid openings are examined

These stopping rules may be revised as data are obtained on the concentration of LA and $PM_{2.5}$ that occur in smoke from the burn chamber.

The number of grid openings needed to achieve the target sensitivity is calculated as follows:

$$GOs = \frac{EFA}{TS \cdot Ago \cdot V \cdot 1000 \cdot F}$$

where:

GOs = Number of grid openings
EFA = Effective filter area (385 mm^2)
TS = Target sensitivity (cc^{-1})
Ago = Area of one grid opening (0.01 mm^2)
V = Volume of flue duct air drawn through the filter = 5 L/min · 15 min = 75 liters
1000 = Conversion from liters to cc
F = Fraction of the primary sample applied to the secondary filter

The optimum value of F is not known. The number of GOs required for various values of F are shown below:

F	GOs
0.1	5
0.01	50
0.001	500

B4.2.2 LA in Impinger Samples

Impinger samples (approximately 200 mL of water) will be mixed thoroughly by shaking and then filtered through 0.2 um MCE filters. LA on filters prepared from impinge fluid will be analyzed using the same basic strategy as described above for LA collected directly on MCE filters. If possible, these filters should be analyzed directly. However, it is expected that these filters will be too heavily loaded to allow direct analysis, and that indirect preparation of the impinger filters may be required.

If so, the direct filter should ashed with low temperature ashing, and the ashed residue suspended in 100 mL of deionized water and sonicated. Then, a series of filters are prepared using a range of aliquot sizes (e.g., 30 mL, 10 mL, 3 mL, 1 mL) to provide a range of filter loadings. The analyst will determine which filter is best for analysis, and utilize that filter. Other filters will be held in archive for use if needed.

The target sensitivity for impinger filter samples is calculated as follows:

$$TS_{if} = TS_{flue} \frac{V_{air}}{V_{if}}$$

where:

TS_{if} = Target sensitivity for impinger fluid (cc^{-1})
TS_{flue} = Target sensitivity for LA in exhaust flue gas (1.2 cc^{-1})
V_{air} = Volume of flue duct air passed through the impinger (5 L/min · 60 min = 300 L)
V_{if} = Volume of impinger fluid (200 cc = 0.2 L)

Based on these values, the target sensitivity for impinge samples is:

TS_{if} = 1.2 cc^{-1} · (300 L/ 0.2 L) = 1,800 cc^{-1}

B4.3 Analysis of LA in Ash

LA in ash material generated by burning duff shall be analyzed for LA in basic accordance with SOP DUFF-LIBBY-OU3 (Rev. 0). Sample preparation and analysis should begin with Step 6.2 of the SOP.

B4.5 Analytical Turn-Around Time for LA Samples

Analytical turn-around time for asbestos shall be negotiated between EPA and the laboratory at the time the samples are shipped. In general, turn-around times of 2-4 weeks are acceptable, but this may be revised as determined necessary by EPA.

B4.6 PM2.5 in Exhaust Flue Smoke

The mass of $PM_{2.5}$ material emitted in smoke during the burn will be measured gravimetrically in general accord with the approach described in EPA Method 201A – *Determination of PM_{10} and $PM_{2.5}$ Emissions from Stationary Sources (Constant Sampling Rate Procedure)*.

B4.7 Measurement of Other Burn-Related Parameters

Table B1 lists other measurements that will be performed during each burn, along with performance criteria. These measurements are important to characterizing each burn event so that results can be properly interpreted and extrapolated to field conditions. These measurements are routinely included in studies of this type, and are not unduly expensive to collect.

Table B1. Performance Criteria for Critical Measurements

Measurement Parameter	Sampling Method(s)	Sub-parameter	Analysis Method	Acceptance Criteria (%Bias/Recovery)	Completeness
Burn Hut Exhaust Velocity Traverses	EPA Method 1A	N/A	N/A	N/A	100%
Burn Hut Exhaust Volumetric Flow Rate	EPA Method 2C (to be performed in conjunction with M5/202, M0010)	Pitot tube leak check	Manometer	± 10% of actual value	100%
		Gas temperature	K-Type Thermocouple	± 3 °F	
Burn Hut Exhaust Moisture Content	EPA Method 4 (to be performed in conjunction with M5/202, M0010)	Post-test meter calibration check	Standard Meter Comparison	± 5 % of pre-calibration	100%
		Balance calibration check	Gravimetric S-Class weights	± 0.5g	100%
Burn Hut Exhaust CO_2/O_2	EPA Method 3A	Calibration error	Instrumental Calibration Gases	± 2%	90% of Test Periods
		Sampling system bias		± 5%	
		Zero & calibration drift		± 3%	
Burn Hut Exhaust CO	EPA Method 10	Calibration error	Instrumental Calibration Gases	± 2%	90% of Test Periods
		Sampling system bias		± 5%	
		Zero & calibration drift		± 3%	

Measurement Parameter	Sampling Method(s)	Sub-parameter	Analysis Method	Acceptance Criteria (%Bias/Recovery)	Completeness
Burn Hut Exhaust Total Filterable and Condensable Particulate	EPA Method 5/202	Post-test meter calibration check	Standard Meter Comparison	± .5% of pre-calibration	67% (minimum 4 of 6)
		Balance calibration check	Gravimetric S-Class weights	± 0.1g	100%
Burn Temperature	N/A	calibration error	K-type thermocouple	± 5 ºC	85%

Continuous Emission Monitors for Burn Chamber Smoke Samples

Continuous instrumental methods will be employed via the use of continuous emissions monitors (CEMs) to measure concentrations of carbon dioxide (CO_2), oxygen (O_2), carbon monoxide (CO), and total hydrocarbons (THC). These instruments will be operated in accordance with EPA Methods 3A (CO_2/O_2), 10 (CO), 6C (SO_2), and 25A (THC) as prescribed in 40 CFR Part 60, Appendix A. CEM testing will begin 10 to15 minutes prior to test material being fed into the burn chamber and will continue for approximately 30 minutes after last material is fed.

Burn hut exhaust gas samples destined to the CEMs (except the THC monitor) will be conditioned to remove water vapor and particulate matter, which are interfering constituents. The sample gas going to the THC monitor will be heated and maintained at 250-300 ºF and filtered with glass fiber filters. The THC monitor requires the sample to be hot and condensate-free to operate properly, as some components of THC can be removed by condensation of water.

Components of the sampling system in contact with the sample gas are constructed of Type 316 stainless steel or Teflon® to minimize the possibility of surface chemical reactions, which can affect the accuracy of the measurements. The sampling manifold will be decontaminated between burns. The CO_2/O_2, THC, and CO sample collection and conditioning system consists of a heated probe and a particulate filter, followed by a moisture-removal trap and an out-of-stack secondary particulate filter. A sample pump (such as Thomas Model 2107CA 18-TFE) transports the effluent sample through a distribution manifold to the analyzers. The configuration of the sampling system allows the calibration gases to be injected either directly to the analyzers or through the complete sample collection and conditioning system.

The concentration signal outputs from the CEMs are connected to a computer-based data acquisition system (DAS). The DAS uses a portable computer and an analog-to-digital converter. For the purposes of these tests, the data will be logged at 6-second intervals without time averaging data. The functioning of the DAS will be checked by verifying that the indicated signal levels are in agreement with calibrated instruments, such as digital voltmeters, TC readouts, etc. These checks will be performed by the EPA Metrology Laboratory.

All pre-test and post-test calibration procedures are performed as outlined in the specific EPA methods. The operation principles of the analyzers are described in the following subsections. Analyzers with equivalent capability and performance may be substituted for the named models.

Burn Hut Exhaust Gas CO_2/O_2 (EPA Method 3A)

Carbon dioxide and oxygen concentrations will be determined by EPA Method 3A – *Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure)*, as described in 40 CFR Part 60, Appendix A. In Method 3A, a continuous gas sample is extracted from the stack and conveyed to instrumental analyzers for the determination of oxygen and carbon dioxide concentration. Results are used in the calculation of sampling duct gas molecular weight.

Burn Hut Exhaust Gas CO (EPA Method 10)

Carbon monoxide emissions will be determined by EPA Method 10 – *Determination of Carbon Monoxide Emissions from Stationary Sources*, as described in 40 CFR Part 60, Appendix A. In Method 10, a continuous gas sample is extracted from the sampling duct and conveyed to an instrumental analyzer (nondispersive infrared sensor [NDIR] or equivalent) for the determination of carbon monoxide concentration. Flow data from concurrent EPA Methods 1A and 2C will be used to calculate carbon monoxide mass emission rates.

Burn Hut Exhaust Gas THC (EPA Method 25A)

EPA Method 25A – *Determination of Total Gaseous Organic Concentration Using a Flame Ionization Analyzer* is applicable over a wide range of THC concentrations, from percent levels down to low parts per million (ppm) levels. The method does not differentiate the species that constitute total hydrocarbons (i.e. methane and non-methane organic compounds [NMOCs] are measured together and reported as one concentration as equivalent propane). Note: Method 25 is specifically designed to measure NMOCs. However, it is not suitable for measuring concentrations that are less than 50 ppm and will not be used.

In Method 25A, a gas sample is extracted from the source through a heated sample line, if necessary, and a glass fiber filter; it is then introduced to a flame ionization analyzer. Results are reported as volume concentration equivalents (ppm by volume) of the calibration gas (propane) or as carbon equivalents. The mass emission rate is calculated by the incorporation of results of EPA Methods 1A and 2C volumetric flow data along with moisture and molecular weights determined by EPA Methods 3A and 4.

Burn Hut Exhaust Gas Volumetric Flow Rate (EPA Methods 1A & 2C)

Flue gas volumetric flow rates will be determined by EPA Method 1A – *Sample and Velocity Traverses for Stationary Sources with Small Stacks or Ducts* and EPA Method 2C – *Determination of Stack Gas Velocity and Volumetric Flow Rate in Small Stacks and Ducts (Standard Pitot Tube)*, as described in 40 CFR Part 60, Appendix A. A measurement location in the effluent stream is selected to minimize angular and cyclonic flow. For these tests, it will be 5 feet downstream of the Burn Hut exhaust duct inlet.

Using Method 1A, the duct cross section is divided into an appropriate number of equal areas and the probe is marked to signify the velocity traverse points. Due to the potential for flow disturbance in small stacks, the sample extraction and flow measurement are performed apart from one another. Sampling ports for extractive samples are located eight equivalent diameters upstream of the velocity sampling ports to allow for the re-establishment of flow stability. Using Method 2C, a traverse for velocity head and sampling duct gas temperature is performed using a standard pitot tube and thermocouple probe to minimize flow disturbance. Sampling duct gas volumetric flow rate is calculated by use of the resultant data, the sampling duct gas density, and duct cross sectional area. Measurements will be performed in conjunction with each test run for filterable/condensable particulate. Flow data, along with pollutant concentration data from concurrent methods, will be used to calculate pollutant mass emission rates.

Burn Hut Exhaust Gas Molecular Weight and Moisture (EPA Methods 3A & 4)

Sampling duct gas molecular weight and diluent concentration will be determined by EPA Method 3A – *Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure)* and EPA Method 4 – *Determination of Moisture Content in Stack Gases*, as described in 40 CFR Part 60, Appendix A. In Method 3A, a gas sample is continuously extracted from the sampling duct and conveyed to instrumental analyzers for the determination of O_2 and CO_2 concentration. Diluent gas concentration and molecular weight are calculated from these results. In Method 4, a gas sample is extracted from the source with moisture being removed and determined gravimetrically and/or volumetrically. Method 4 samples will be taken as a part of the EPA Method 5/202, and M0010 samples.

Burn Hut Exhaust Particulate Matter ($PM_{2.5}$) in Smoke

Air from the exhaust flue will be sampled continuously during the burn event to estimate the average concentration of particulate matter in the exhaust smoke (ug/m^3). Because the results will be coupled with USFS models that predict particulate concentrations in terms of $PM_{2.5}$, the sampling device will be designed to collect only $PM_{2.5}$, not total particulate matter.

B5. QUALITY CONTROL

The quality control (QC) requirements for TEM analyses of asbestos in smoke samples or other media (duff, ash) are patterned after the requirements set forth by NVLAP. There are three types of laboratory-based QC analyses that are performed for TEM. Each of these is described below.

Lab Blank - This is an analysis of a TEM grid that is prepared from a new, unused filter in the laboratory and is analyzed using the same procedure as used for field blank samples.

Recounts - A recount is an analysis where TEM grid openings are re-examined after the initial examination. The type of recount depends upon who is performing the re-examination. A *Recount Same* (RS) describes a re-examination by the same microscopist who performed the initial examination. A *Recount Different* (RD) describes a re-examination by a different microscopist within the same laboratory than who performed the initial examination. An *Interlab* (IL) describes a re-examination by a different microscopist from a different laboratory.

Repreparation - A repreparation is an analysis of a TEM grid that is prepared from a new section of filter as was used to prepare the original grid(s). Typically, this is done within the same laboratory as did the original analysis, but a different laboratory may also prepare grids from a new piece of filter.

For this project, the frequency of these laboratory-based QC samples will be as follows:

QC Sample Type	QC Sample Rate
Lab Blank	1% (1 per 100)
Recount Different	2% (1 per 50)
Interlab	2% (1 per 50)
Repreparation	2% (1 per 50)

The list of samples for Recount Different, Interlab, and Repreparation will be selected by EPA and provided to the laboratory by the EPA project manager after the results of the original sample analyses have become available.

The most recent version of laboratory modification LB-000029B (see Appendix B) summarizes the acceptance criteria and corrective actions for TEM laboratory QC analyses that will be used to assess data quality.

B6/B7. INSTRUMENT MAINTAINANCE AND CALIBRATION

Field Instruments

The only field instrument that will be utilized in this project is a scale capable of weighing duff, both the bulk amount collected (approximately 100 pounds), and for packaging (about 0.5 pounds per bag). Before use in the field, any scales used for this project will be inspected to ensure the scales are in proper working order, and shall be checked for accuracy by weighting objects of known weight. This calibration/verification shall be documented in the field log book.

Burn Facility Instruments and Equipment

Maintenance and calibration of equipment needed to collect samples and flue gas measurements during burn studies are detailed in Appendix A.

Laboratory Instruments

All TEM instrument used for this project will be maintained and calibrated in accordance with the manufacturer's instructions. If any deficiencies in instrument function are identified, all analyses shall be halted until the deficiency is corrected. The director of the analytical laboratory shall maintain a log that documents all routine maintenance and calibration activities, as well as any significant repair events, including documentation that the deficiency has been corrected.

B8. INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Field Supplies

There are no acceptance requirements for consumable field supplies used in this project.

Laboratory Supplies

The laboratory manager is responsible for ensuring that all reagents and disposable equipment used in this project is free of asbestos contamination. This is demonstrated by the collection of laboratory blank samples, as described in Section B5.

B9. NON-DIRECT MEASUREMENTS

There are no non-direct measurements that are anticipated for use in this project.

B10. DATA MANAGEMENT

Data Management Scheme

Data generated during this project consists of paired measurements of LA and $PM_{2.5}$ in samples of smoke from OU3 duff burned under various conditions. All analytical data generated will be transmitted to EPA's contractors for verification and evaluation.

Data Deliverables

LA Data

Asbestos data generated during this project will be transmitted from the analytical laboratory in the form of Libby-specific EDD spreadsheets. Analytical results will include the structure-specific data for all TEM analyses. All data entry will be reviewed and validated for accuracy by the laboratory data entry manager or appointed delegate before transmittal.

All asbestos EDDs will be transmitted to EPA's data management contractor (CDM) electronically. Whenever possible, data files should be transmitted by posting the file to the project-specific eRoom (https://team.cdm.com/eRoom/) in the "Laboratory EDDs" folder. This eRoom will have controlled access (i.e., user name and password are required) to ensure data access is limited to appropriate project-related laboratory personnel.

If files are too large to post to the eRoom, they should be provided on a compact disc to the following address:

Attn: Lynn Woodbury
CDM
555 17th Street, Suite 1100
Denver, CO 80202
303-383-2382

$PM_{2.5}$ Data

$PM_{2.5}$ data generated during this project will be transmitted using RTP's standard reporting EDD for $PM_{2.5}$. All EDDs will be transmitted using the same procedures as for asbestos EDDs described above.

Data Management Applications

All data generated as part of this project sampling will be maintained in a Burn Study-specific Microsoft® Access database. This will be a relational database with tables designed to store information on burn conditions, time of sample collection, attributes of the burn material, sample collection method, preparation and analysis details, and analytical results. Results will include all asbestos data, including detailed structure attributes for all LA structures observed in the TEM analyses, with paired $PM_{2.5}$ data.

EPA staff and designated contractors will be responsible for data analysis and reduction, including tabular and graphical data summaries and risk calculations.

Database Administrators

Day-to-day operations of the study database will be under the control of EPA contractors. The primary database administrator will be responsible for sample tracking, uploading new data, performing error checks, and making any necessary data corrections. New records will be added to the study database within an appropriate time period of sample and/or EDD receipt.

Incremental backups of the study database will be performed daily Monday through Thursday, and a full backup will be performed each Friday. The full backup tapes will be stored off-site for 30 days. After 30 days, the tape will be placed back into the tape library to be overwritten by another full backup.

Each Friday, a copy of the study database will be posted to a project-specific FTP site to allow timely access to results by data users. The study database posted to the FTP site will include the post date in the file name (e.g., MasterOU3DB_20090831.mdb).

Data Storage

All original data records (both hard copy and electronic) will be cataloged and stored in their original form until otherwise directed by the EPA RPM. At the termination of this study, all original data records will be provided to the EPA RPM in a format specified by EPA for incorporation into the OU3 project files.

C1. ASSESSMENT AND RESPONSE ACTIONS

Assessments and oversight reports to management are necessary to ensure that procedures are followed as required and that deviations from procedures are documented. These reports also serve to keep management current on field activities. Assessment, oversight reports, and response actions are discussed below.

Field Oversight

All individuals who collect samples during field activities will be provided a copy of this QAPP and will be required to participate in a pre-sampling readiness review meeting to ensure that methods and procedures called for in this QAPP and associated SOPs are understood and that all necessary equipment is on hand. EPA may perform random and unannounced field audits of field sampling collection activities, as may be deemed necessary. The EPA field auditor has the authority to direct changes in field activities, or to halt field activities if needed until a remedy to an unexpected problem can be identified.

Laboratory Oversight

All laboratories selected for analysis of samples for asbestos will be part of the Libby analytical team for OU3. These laboratories have all demonstrated experience and expertise in analysis of LA in environmental media, and all are part of an on-going site-specific quality assurance program designed to ensure accuracy and consistency between laboratories. These laboratories are audited by EPA and NVLAP on a regular basis. Additional laboratory audits may be conducted upon request from the EPA, as may be needed.

Response Actions

If any inconsistencies or errors in field or laboratory methods and procedures are identified, response actions will be implemented on a case-by-case basis to correct quality problems. All response actions will be documented in a memo to the EPA RPM for OU3 at the following address:

Christina Progess
U.S. EPA, Region 8
1595 Wynkoop Street
Denver, CO 80202-1129
E-mail: progess.christina@epa.gov

Any problems that cannot be corrected quickly through routine procedures may require implementation of a corrective action request (CAR) form.

C2. REPORTS TO MANAGEMENT

No regularly-scheduled written reports to management are planned as part of this project. However, field and analytical staff will promptly communicate any difficulties or problems in implementation of the QAPP to EPA, and may recommend changes as needed. If any revisions to this QAPP are needed, the EPA RPM will approve these revisions before implementation by field or analytical staff.

Once all project-related activities are completed, EPA contractors will prepare a report that summarizes the work that was performed, presents the data collected, and provides a summary and interpretation of the findings. This will be submitted to EPA for review, and revised as directed by the EPA RPM.

D1. DATA REVIEW, VERIFICATION AND VALIDATION

Acceptance Criteria for LA Measurements

Several factors are considered in determining the acceptability of LA measurements in samples analyzed by TEM. This includes the following:

1. Evenness of filter loading. This is evaluated using a chi-square test, as described in ISO 10312 Annex E. If a filter fails the chi-square test for evenness, the result may not be representative of the true concentration in the sample, and the result should be given low confidence.

2. Results of LA QC samples. This includes laboratory blank samples, as well as various types of recount and repreparation analyses. If significant LA contamination is detected in laboratory blanks, all samples prepared on that day should be considered to be potentially biased high. If agreement between original analyses and repreparation or recount analyses is poor, results for those samples should be given low confidence.

Acceptance Criteria for $PM_{2.5}$ Measurements

Acceptance criteria for $PM_{2.5}$ measurements are described in Appendix A.

D2. VERIFICATION AND VALIDATION METHODS

Data validation consists of examining the sample data package(s) against pre-determined standardized requirements. The validator may examine, as appropriate, the reported results, QC summaries, case narratives, COC information, raw data, initial and continuing instrument calibration, and other reported information to determine the accuracy and completeness of the data package. During this process, the validator will verify that the analytical methodologies were followed and QC requirements were met. The validator may recalculate selected analytical results to verify the accuracy of the reported information. Analytical results will then be qualified as necessary.

Data verification includes checking that results have been transferred correctly from laboratory data printouts to the laboratory report and to the EDD. In general, field and analytical results will be performed at a frequency of 10%. This initial rate may be increased if errors are detected. Data validation, review, and verifications must be performed on sample results before distribution to the public for review.

EPA staff or technical contractors will be responsible for implementing all data verification and validation activities. If errors or suspected errors in the data are identified, EPA shall contact the analytical laboratory to request a double check of data entry and the implementation of corrections to the data, as may be appropriate.

D3. RECONCILIATION WITH USER REQUIREMENTS

Once all samples have been collected and analytical data has been generated, data will be evaluated to determine if study objectives were achieved. Evaluation of the data for this project will include a qualitative and quantitative review of all QC samples and all deviations from designs and procedures described in this report, along with conclusions regarding the reliability of the data for their intended use. Any limitations in data reliability will be provided to the chief data users in a written report, so that these limitations may be accounted for when applying or using the data.

E. REFERENCES

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Rohs AM, Lockey JE, Dunning KK, Shulka R, Fan H, Hilbert T, Borton E, Wiot J, Meyer C, Shipley RT, LeMasters GK, Kapol V. 2007. Low level Fiber Induced Radiographic Changes Caused by Libby Vermiculite: A 25 year Follow-up Study. *Am J Respiratory and Critical Care Medicine.* Published online December 6, 2007 as doi:10.1164/rccm.200706-814OC.

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FIGURE A1. ORGANIZATIONAL CHART

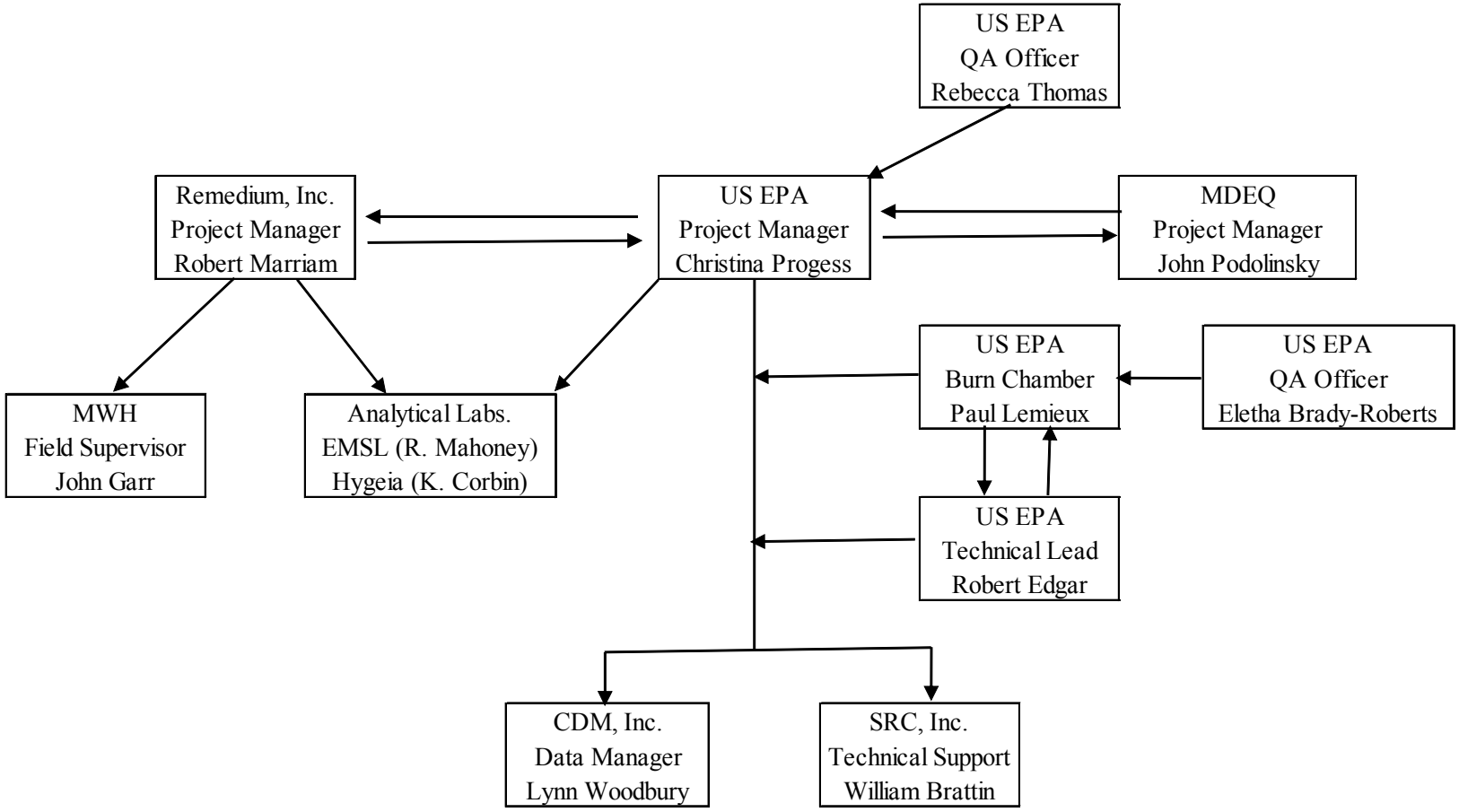
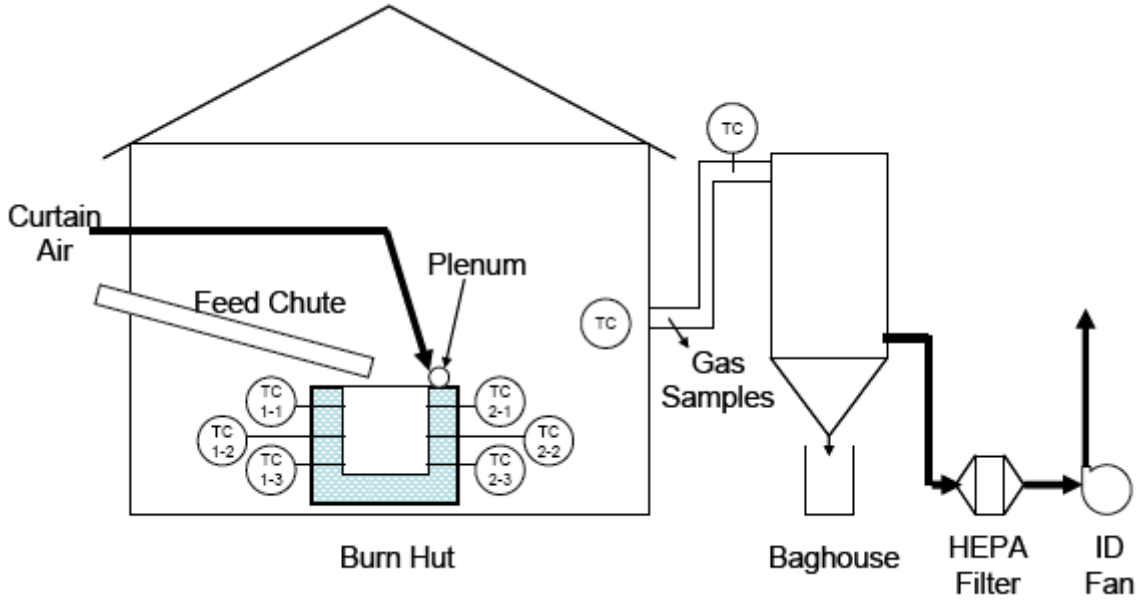


FIGURE B1.
BURN CHAMBER SCHEMATIC



Source: Lemieux et al. 2011

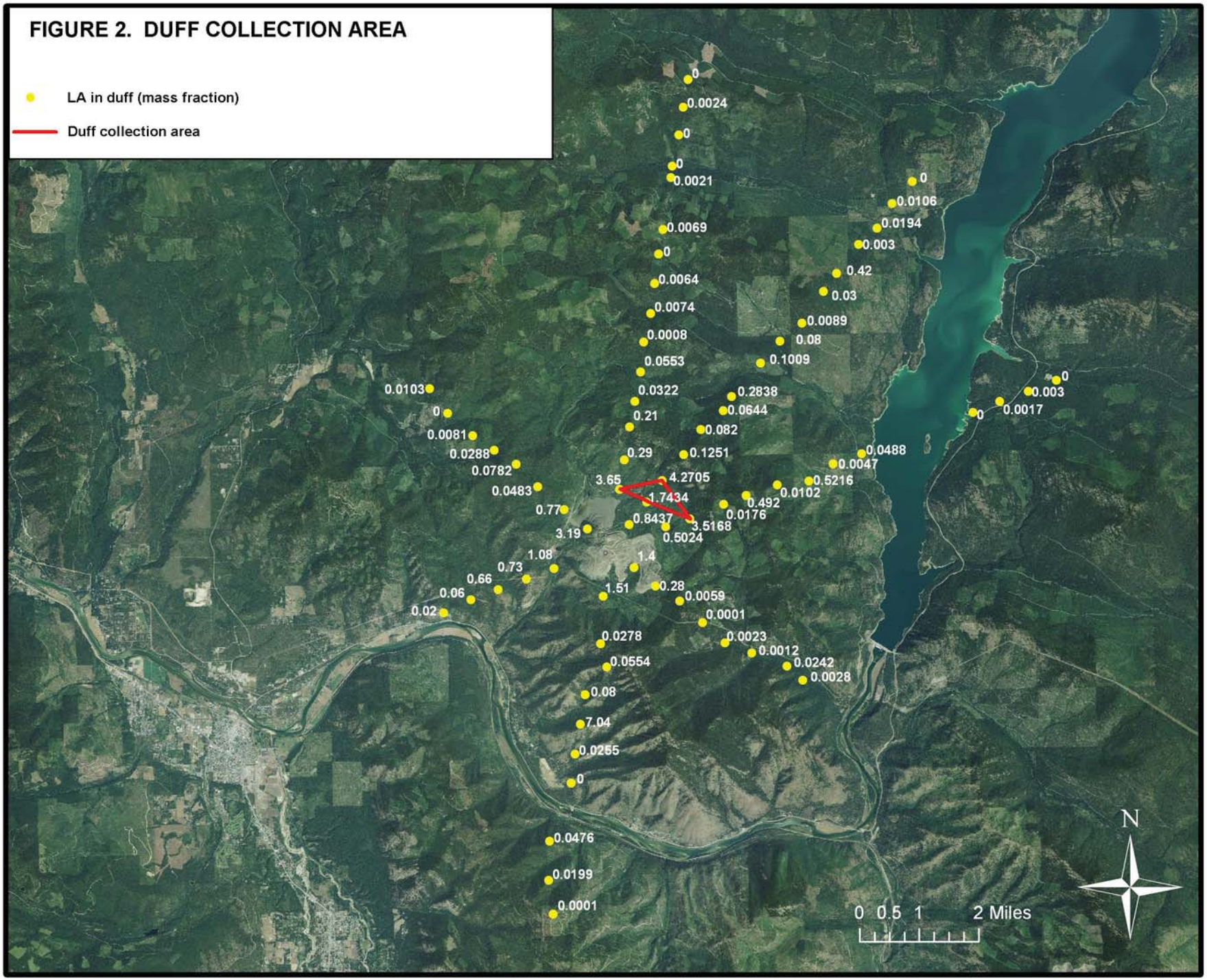


FIGURE 2. DUFF COLLECTION AREA
LA in duff (mass fraction)
Duff collection area
0
0.0024
0
0
0.0021
0
0.0106
0.0194
0.003
0.0069
0
0.42
0.0064
0.03
0.0074
0.0089
0.0008
0.08
0.0553
0.1009
0
0.003
0.0017
0
0.0103
0.0322
0.2838
0
0.0644
0.21
0.082
0.0081
0.0288
0.29
0.1251
0.0488
0.0047
0.0782
3.65
4.2705
0.5216
0.0483
1.7434
0.0102
0.492
0.77
0.0176
0.8437
3.5168
3.19
0.5024
1.08
1.4
0.73
0.66
1.51
0.28
0.06
0.0059
0.02
0.0001
0.0278
0.0023
0.0012
0.0554
0.0242
0.0028
0.08
7.04
0.0265
0
0.0476
0.0199
0.0001
N
0 0.5 1 2 Miles

ATTACHMENT A

CALCULATION OF CANCER RISK-BASED CONCENTRATION OF LA IN BREATHING ZONE AIR OF FIREFIGHTERS

1.0 BASIC EQUATIONS

Excess cancer risk from inhalation exposure to LA in air is calculated as follows (EPA 2008):

$$Risk = EPC \cdot TWF \cdot IUR_{a,d}$$

where:

EPC = Long-term average concentration of LA in breathing zone air (PCME f/cc)
TWF = Time weighting factor (the fraction of full time that exposure occurs)
$IUR_{a,d}$ = Inhalation unit risk for an exposure that begins at age "a" (years) and lasts for duration "d" (years)

Rearranging this equation yields:

$$RBC = Target\ Risk / (TWF \cdot IUR_{a,d})$$

where:

RBC = Risk based concentration (PCME f/cc)

2.0 RBC FOR FIREFIGHTERS

The Target Risk is set to a value of 1E-05.

The value of TWF is calculated as follows:

$$TWF = ET/24 \cdot EF/365$$

where:

ET = exposure time (hours/day)

EF = exposure frequency (days/year)

For USFS firefighters, the following values are assumed for firefighting activities in OU3:

ET = 16 hours/day
EF = 10 days/year

Based on this, TWF = (16/24) · (10/365) = 0.0183

The value of $IUR_{a,d}$ depends on the age at first exposure (a) and the duration of exposure (d). For USFS firefighters, the following values are assumed for firefighting activities in OU3:

a = 20 years
d = 30 years

Based on this, $IUR_{a,d}$ is 0.0748 $(PCME\ f/cc)^{-1}$ (EPA 2008).

Applying these exposure assumptions, the RBC for firefighters is calculated as follows:

RBC = 1E-05 / (0.0183 · 0.0748) = 0.0073 PCME s/cc

APPENDIX A

DETAILED QAPP FOR ACTIVITIES AT THE BURN CHAMBER FACILITY

[pending completion by RTP]

APPENDIX B

STANDARD OPERATING PROCEDURES

DUFF-LIBBY-OU3 (Rev 0)
EPA-LIBBY-08 (Rev 1)
OU3 SOP No. 8 (Rev 2)

Date: February 7, 2008

SOP DUFF-LIBBY-OU3 (Rev. 0)

Title: SAMPLING AND ANALYSIS OF DUFF FOR ASBESTOS

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager	Mary Goddard for Bonnie Project Chemist Bonita Lavelle, USEPA RPM	2/7/08
SOP Author	W J Brattin William Brattin, SRC	2/7/08

Revision No.	Date	Reason for Revision
0	02/07/2008	--

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for collection and analysis of duff samples for Libby amphibole asbestos (LA). Duff consists of the un-decomposed twigs, needles and other vegetation and the layer of partially- to fully-decomposed litter that occurs on top of the mineral soil in forested areas. This procedure will be used by USEPA Region 8 for the Remedial Investigation work for Operable Unit 3 performed at the Libby Asbestos Superfund site.

2.0 RESPONSIBILITIES

The Field Sampling Team Leader is responsible for ensuring that all duff samples are collected in accord with this SOP. The Laboratory Director is responsible for ensuring that duff samples provided to the laboratory for evaluation by this SOP are prepared and analyzed in accord with the requirements of this SOP. It is the responsibility of the Field Sampling Team Leader and the Laboratory Director to communicate the need for any deviations from the SOP with the appropriate USEPA Region 8 Remedial Project Manager or Regional Chemist.

3. 0 EQUIPMENT

3.1 Field Equipment

- Ziploc® plastic bags
- sample identification labels
- GPS unit
- field log book
- field sample data sheet(s)
- ink pen
- clear packaging tape

3.2 Laboratory Equipment /Reagents

- Large aluminum trays
- Drying oven
- Large metal tray(s) (large enough for duff sample to cover bottom up to 1/2 in.)
- Muffle furnace
- Glass stirring rods
- Fume hood
- HEPA filtered hood
- Reagent grade or better acetone
- Reagent grade or better HCl
- Fiber-free deionized water (FDI water)
- Ultrasonic bath, producing a rate of energy deposition in the range of 0.08-0.12 MW/m^3
- Disposable plastic filter funnel apparatus

- Disposable filter funnels with straight sides [VWR # 145-0020]
- Culture dishes [VWR # 25388-581, case of 500]
- 47 mm 0.45 micron MCE or 0.4 micron PC filters
- Kim wipes or alternative paper
- Ziploc plastic bags
- Glass petri dishes
- Glass microscope slides
- Low temperature plasma asher
- Vacuum evaporator (carbon coater)
- Graphite or carbon rods
- HEPA laminar flow hood
- Acetone vapor generator
- Grids
- Fine forceps
- Grid storage boxes
- Jaffe wick or sponge
- Transmission electron microscope with the following capabilities:
  - 100 Kev
  - fine probe size <250 nm
  - Energy Dispersive X-Ray Analysis (EDXA)
  - Selective Area Electron Diffraction (SAED)

4.0 METHOD SUMMARY

A duff sample is collected by hand at a selected field location and placed in a plastic bag. Duff samples are prepared for analysis by high temperature ashing to remove organic matter. The residue is then analyzed for LA by transmission electron microscopy (TEM) and/or by Polarized Light Microscopy (PLM), as specified in the project-specific Sampling and Analysis Plan (SAP).

5.0 SAMPLE COLLECTION

Duff samples should be collected from the soil sampling stations specified in the project-specific SAP. At each specified sampling station, collect any fresh or partially decayed organic debris (e.g., twigs, leaves, pine needles) using a freshly-gloved hand from the soil surface within an area that is approximately 6 in. x 6 in. Care should be taken to ensure that the top layer of soil beneath the organic debris is not included in the duff material sample. Place the duff material into a large, air-tight, re-sealable plastic bag. Label the bag with the same sample identifier as the soil field sample, and place clear packaging tape over the sample identifier label.

Attachment A provides a Field Sample Data Sheet (FSDS) for recording field information on each duff sample. [Note: in some cases, an alternative FSDS may be specified and provided in the project specific SAP]. Note any special circumstances or conditions about the sampling location. Obtain and record the GPS coordinates of the sampling location on the FSDS form.

6.0 SAMPLE PREPARATION AND ANALYSIS

6.1 Drying and Ashing

Weigh and record the tare weight of a clean, dry aluminum tray of approximately quart size. Fill the aluminum tray to approximately ¾ full. The samples may be split across as many trays as may be needed, providing the samples' identification number is clearly marked on each tray. In addition, for tracking purposes each tray should possess a mark to make it unique and identifiable from the other trays. This identifier shall be recorded in the laboratory preparation logs. Each tray will need to be initially tared and then gravimetrically tracked through the process. Place the tray(s) with the sample into a drying oven. Heat to 60°C and hold at this temperature until weight stabilizes (at least 10 hours). Record the dry weight and calculate the mass of the dried duff sample by the difference.

Once samples are dried, they then shall be ashed. Weigh and record the tare weight of one or more clean metal pans capable of withstanding the heat of a 450°C oven. Working under a hood, transfer the dried duff to the tared pan(s), place a lid on the pan and move to a muffle furnace. Ramp up the furnace from a cold start to 450°C and hold at this temperature for 18 hours or until all organic matter is removed.

Allow the pan(s) to cool. Remove the lid(s), weigh and record the mass of the pan(s) plus the ashed residue. Calculate the mass of the ashed residue in each pan by difference. If the sample was ashed in more than one pan, compute the total mass of the ashed residue for the sample by summation across pans.

Under a laminar flow hood, slowly pour the ash from each sample into a Ziploc bag. If the sample was ashed in more than one pan, all the pans for that sample are combined into a single Ziploc bag. If the ash still retains some structure, seal the bag tightly and manipulate the ash by hand to reduce it to a fine homogenous powder. Invert the bag 3-4 times to thoroughly mix the ash.

All information regarding sample preparation shall be recorded using the sample preparation log sheet, presented as Attachment B.

6.2 TEM Analysis

Acid Treatment

Remove an aliquot of approximately 0.25 g of the well-mixed ash and place into a crucible. Record the weight (measured to an accuracy of ± 0.01 g) on the sample preparation data sheet (see Attachment B). To the ashed residue in the crucible, add just enough FDI water (approx 1-2 mL) to cover the surface of the residue. Slowly add concentrated HCl to the wetted ash (approx. 10-20 mL). Typically a visible effervescing is observed. Add the HCl slowly to keep this reaction controlled. A small glass stirring rod is useful at this point to gently stir the ash and expose all material to the acid.

If after 3-5 minutes there is no further visible reaction, proceed to the next step. If bubbling is still occurring, continue observation and gentle stirring for up to an additional 5 minutes.

Dilute the sample by adding FDI water directly to the crucible (approx 20 mL) using a squirt bottle. Pour the sample into an unused disposable 100 mL specimen container with lid. Rinse out any remaining residue from the crucible into the specimen container. Do not exceed 100 mL total volume. Bring the total volume to 100 mL with DI water.

Cap the specimen cup and agitate the sample by inversion 5 or 6 times. Loosen the cap slightly and sonicate for 2 minutes. After sonication, tighten the cap and then dry the exterior of the specimen container with a laboratory wipe.

Filtration

Agitate the sample by inversion 5 or 6 times. Withdraw an initial aliquot of 0.1 to 1 mL of sonicated sample. Transfer this aliquot into a new disposable specimen container with lid. Bring the volume up to approximately 100 mL with FDI water. Cap and agitate by inversion (5 or 6 times).

Filter this entire volume onto a 47 mm mixed cellulose ester (MCE) filter with 0.4 um pore size.

If the filter appears overloaded (overall particulate level > 20%), repeat the process above, selecting a smaller aliquot volume, as suggested by the degree of overloading. Conversely, if the filter looks too lightly loaded, filter a larger aliquot.

After filtration, transfer the filter membranes to individual disposable labeled Petri dishes with lids. With Petri dish covers ajar, gently air dry the filters in a HEPA protected environment.

TEM Examination

Prepare 3 grids for TEM analysis as detailed in International Organization for Standardization (ISO) TEM method 10312, also known as ISO 10312:1995(E). Utilize 2 grids for analysis, holding the third in case of problems. After analysis, archive all three grids for potential future reanalysis.

Counting rules

Examine the grids using TEM in accord with ISO 10312 and all relevant Libby site-specific modifications, including the most recent version of LB-000016, LB-000019, LB-000028, LB-000029, LB-000029a, LB-000030, LB-000053, and LB-000066. All fibrous amphibole structures that have appropriate Selective Area Electron Diffraction (SAED) patterns and Energy Dispersive X-Ray Analysis (EDXA) spectra, and having length greater than or equal to 0.5 um and an aspect ratio (length: width) $\geq$ 3:1, will be recorded on the Libby site-specific laboratory bench sheets and electronic data deliverable (EDD) spreadsheet for TEM analysis of duff samples. Data recording for chrysotile (if observed) is not required.

Stopping rules

The target analytical sensitivity for sample analysis should be specified in the SAP. In the absence of a project-specific target sensitivity, the default sensitivity should be 1E+07 $(grams)^{-1}$, which is likely to correspond to a mass fraction of less than about 0.005 grams asbestos per gram duff (dry wt). The analytical sensitivity is calculated using the following equation:

$$S = \frac{EFA}{GO \cdot Ago \cdot Mass \cdot F}$$

where:

S	=	Sensitivity (1/g dry wt)
EFA	=	Effective filter area (mm^2)
GO	=	Number of grid openings counted
Ago	=	Area of one grid opening (mm^2)
Mass	=	Mass of the dried (but not ashed) duff sample (g)
F	=	Fraction of the starting duff sample applied to the filter

Count the sample until one of the following occurs:

- The target sensitivity is achieved.
- A total of 50 or more LA structures are observed. In this case, counting may cease after completion of the grid opening that contains the 50^{th} LA structure.
- A total of 100 grid openings are counted without reaching the target sensitivity or observing 50 LA structures. In this event, the analysis should stop after completion of the 100^{th} grid opening.

TEM Data Deliverable

All data on the number, type and size of LA fibers observed during TEM analysis in the laboratory will be provided as an electronic data deliverable (EDD) using the most recent version of the spreadsheet developed for this purpose ("TEM Duff.xls"). The results for each sample will be expressed in terms of LA fibers per gram duff (dry weight), and also in terms of grams of LA per gram of duff (dry weight).

6.3 PLM Analysis

If analysis by PLM is called for in the project-specific SAP, the analysis will be performed on an aliquot of the ashed and homogenized residue using method PLM-VE as detailed in the most recent version of SOP SRC-LIBBY-03. PLM-VE is a semi-quantitative analytical method for asbestos that utilizes Libby-specific reference materials to allow assignment of samples into one of four "bins", as follows:

- Bin A (ND): non-detect
- Bin B1 (Trace): LA detected at levels lower than the 0.2% reference material
- Bin B2 (<1%): LA detected at levels lower than the 1% reference material but higher than the 0.2% reference material
- Bin C: LA detected at levels greater than or equal to 1%

A potential limitation to this approach is that the site-specific reference materials are based on LA in soil, not LA in ashed residue. This may introduce additional uncertainty into the results, but no reference materials based on ashed residue are presently available.

PLM-VE results will be recorded using the most recent version of the Libby site-specific EDD spreadsheet for PLM-VE analysis ("PLM (VE & PC) Data Sheet and EDD.xls").

7.0 QUALITY ASSURANCE

7.1 Field-Based Quality Assurance

Field Duplicates

Field duplicate duff samples will be collected at a frequency specified in the project-specific SAP. In the absence of such specification, the rate should be no less than 5%. Each field duplicate should be collected from a location close to the primary sample, and from an area of approximately equal size. Field duplicate samples should be labeled with a unique identifier. Sample details should be recorded on the appropriate soil FSDS, including the unique identifier of the "parent" field sample. Field duplicates are used to evaluate the sampling and analysis variability across duff samples. Unless indicated differently in the project-specific SAP, samples will not be qualified purely as a result of the difference between measured values between original and duplicate pairs.

7.2 Laboratory-Based Quality Assurance for TEM Analyses

Drying Blanks

For the purposes of this analysis, a drying blank will consist of one clean aluminum pan placed empty into the drying oven along with pans containing field samples of duff. After drying the duff samples, the clean tray will be removed and the surface will be rinsed with about 100 mL of FDI water into a clean container, which in turn will be filtered and prepared for TEM analysis. Detection of fibers on the drying blank filter will be taken as an indication of potential cross-contamination during drying.

Drying blanks should be prepared at a rate specified in the project-specific SAP. In the absence of a project-specific specification, drying blanks should be prepared at a rate of one per day that drying of samples is occurring. Unless indicated differently in the project-specific SAP, if the drying blank reports LA fibers, all samples in that drying batch will be assigned a qualifier to indicate the potential for cross-contamination.

Laboratory Blanks

A laboratory blank is a filter that is prepared by processing a clean crucible in the same way that a duff sample is prepared. That is, a clean crucible is treated by addition of FDI water and HCl, as described above. The contents of the crucible are then rinsed out, diluted to 100 mL, and an aliquot at least as large as the highest volume aliquot for the sample set is removed and used to prepare a filter for TEM examination. This type of blank is intended to indicate if contamination is occurring at any stage of the sample preparation procedure.

Laboratory blanks should be prepared at a rate specified in the project-specific SAP. In the absence of a project-specific specification, laboratory blanks should be prepared at a rate of 3%. Unless indicated differently in the project-specific SAP, if the laboratory blank reports LA fibers, all samples in that analytical batch will require re-preparation.

Filtration Blanks

A filtration blank is a clean filter that is prepared by passing 100 mL of laboratory FDI water through it. The purpose of this type of blank is to ensure that the filters are not contaminated in the laboratory, and that fluids used for diluting and processing samples are fiber-free.

Filtration blanks should be prepared at a rate specified in the project-specific SAP. In the absence of a project-specific specification, filtration blanks should be prepared at a rate of 2%. Unless indicated differently in the project-specific SAP, if the laboratory blank reports LA fibers, all samples in that analytical batch will require re-preparation.

Laboratory Duplicates

Laboratory duplicates will be prepared by applying a second aliquot of ashed residue suspension to a new filter, which is then prepared and analyzed in the same fashion as the original filter. The frequency of laboratory duplicates should be specified in the project-specific SAP. In the absence of such specification, the rate should be no less than 5%. Unless indicated differently in the project-specific SAP, samples will not be qualified purely as a result of the difference between measured values between original and duplicate pairs.

Recounts

The precision of TEM sample results should be evaluated by recounting selected grid openings in accord with the requirements specified in the most recent version of LB-000029.

7.3 Laboratory-Based Quality Assurance for PLM-VE Analyses

Laboratory Duplicates

Laboratory duplicate PLM-VE analyses will be prepared by examining a second aliquot of ashed and homogenized residue. The frequency of laboratory duplicates should be specified in the project-specific SAP. In the absence of such specification, the rate should be no less than 5%.

Unless indicated differently in the project-specific SAP, samples will not be qualified purely as a result of the difference between measured values between original and duplicate pairs.

8.0 REFERENCES

International Organization for Standardization. 1995. Ambient Air – Determination of asbestos fibres – Direct-transfer transmission electron microscopy method. ISO 10312:1995(E).

Phipps, R.L. 1985. Collecting, Preparing, Cross-dating and Measuring Tree Increment Cores. U.S. Geological Survey Water Resources Investigations Report 85-4148

Ward TJ, T Spear, J Hart, C Noonan, A Holman, M Getman, and JS Webber. 2006. Trees as Reservoirs for Amphibole Fibers in Libby, Montana. Science of the Total Environment 367: 460-465.

ATTACHMENT A

FIELD SAMPLING DATA SHEET (FSDS)

Sheet No.: Duff- _____

LIBBY FIELD SAMPLE DATA SHEET (FSDS) rev0
DUFF

Field Logbook No: _____ Page No: _____
Station ID: _____ Sampling Date: _____
GPS Coordinate System: _____ Elevation Coordinate System: _____
X coordinate:_____ Y coordinate:_____ Elevation:_____
Sampling Team: _____ Sampler Initials: _____
Station Comments: _____

Data Item	Sample 1	Sample 2	Sample 3
Index ID (place pre-printed label in field provided)			
Sample Time (hh:mm)			
Sample Type (circle one):	Grab Composite # of Composites: _____	Grab Composite # of Composites: _____	Grab Composite # of Composites: _____
Field QC Type (circle one):	FS (field sample) FD (field duplicate) For FD, Parent ID: _____	FS (field sample) FD (field duplicate) For FD, Parent ID: _____	FS (field sample) FD (field duplicate) For FD, Parent ID: _____
Field Comments:			
Entered by (Provide initials):		**Validated by (Provide initials):**	

For Data Entry Completion (Provide Initials)	Completed by	QC by

ATTACHMENT B

DUFF PREPARATION SAMPLE DATA SHEET (PSDS)

LIBBY DUFF PREPARATION SAMPLE DATA SHEET (PSDS)

Laboratory Name: _____ Lab Job No.: _____ Lab QC Batch No.: _____ SOP: DUFF-LIBBY-OU3 (Rev 0)

Preparation by: _____ Preparation Date: _____

Drying Oven Temp. (°C): _____ Muffle Furnace Temp. (°C): _____ HCL Reagent Tracking No: _____

	SAMPLE INFORMATION			DRYING						ASHING				FILTER PREP			
	Index ID	Lab Sample ID	Mass (g), as received	Tray ID(s) used in drying	Tray weight (g)	Mass (g), during drying [tray + sample]			Mass (g), after drying [sample only]	Pan ID(s) used in ashing	Pan weight (g)	Mass (g), after ashing [pan + sample]	Mass (g), after ashing [sample only]	Mass of ash (g) taken for analysis	Volume of HCl added (mL)	Aliquot volume (mL)	Notes
						Check 1	Check 2	Check 3									
Example	X-12345	026589	500.3	A	5.71	63.12	55.90	55.84	50.13	A	15.87	36.98	21.11	0.26	15.7	1.0	
	↓	↓	↓	B	4.99	70.56	63.02	63.11	58.12	B	16.20	44.05	27.85				
	↓	↓	↓	C	5.23	89.63	71.85	72.03	66.8								

Note: All mass measurements should be recorded to an accuracy of ± 0.01 g.

QA Check by: Date:

Libby Standard Operating Procedure
Indirect Preparation of Air and Dust Samples for TEM Analysis
Approved for Use at the Libby Superfund Site Only

Date: 1/23/07 SOP No. EPA-LIBBY-08

Title: INDIRECT PREPARATION OF AIR AND DUST SAMPLES FOR TEM ANALYSIS

Author: Ron Mahoney, Ed Cahill EMSL Analytical, Inc.

SYNOPSIS: A standardized method is presented for indirect preparation of air and dust samples for analysis by TEM.

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8		1/23/07

REVISION LOG

Revision	Date	Reason
0	11/28/06	--
1	1/23/07	Clarification of filter configuration, secondary and tertiary dilution procedures.

1.0 PURPOSE

Some air samples collected at the Libby Superfund site are overloaded with debris and/or have obvious non-uniform loading, so analysis for asbestos by Transmission Electron Microscopy (TEM) requires an indirect preparation of the sample. All dust samples collected at the Libby Superfund site are prepared for TEM analysis using an indirect preparation. The purpose of this SOP is to provide a standardized procedure for the indirect preparation of air and dust samples that minimizes the loss of sensitivity. In addition, this SOP allows for the retention of a portion of the original air sample filter for archive whenever possible.

2.0 RESPONSIBILITIES

The Laboratory Director is responsible for ensuring that all laboratories participating in the analysis of air samples at the Libby site are aware of this SOP and that all analysts follow this SOP. Laboratory managers and analysts are responsible for communicating to the Libby laboratory coordinator (CDM), Volpe Center and appropriate USEPA Region 8 Remedial Project Manager or Regional Chemist any recommended changes or proposed improvements to the SOP.

3.0 EQUIPMENT

Equipment needed to perform indirect preparations of air samples includes the following:

- Transmission electron microscope (NVLAP compliant)
- Energy dispersive X-ray system (NVLAP compliant)
- High vacuum carbon evaporator with rotating stage
- HEPA hood (NVLAP compliant)
- Exhaust or fume hood
- Particle-free water
- Glass container for ashing
- Disposable single use containers of at least 100 ml capacity
- Waterproof marker
- Forceps
- Ultrasonic bath
- Appropriate disposable glass or variable pipets with disposable tips
- Disposable 25 mm filter funnels
- Side arm filter flask
- Cellulose support pad, 25 mm diameter
- MCE filters, 25 mm diameter, $\leq$ 0.22 µm and 5.0 µm pore size
- Storage container for 25 mm filter
- Glass slides, approximately 25 x 76 mm in size
- Scalpel blades, # 10 or equivalent and handle
- Desiccator or low temperature drying oven
- Acetone, reagent grade
- Glacial acetic acid

- Plasma asher, low temperature
- pH paper
- Tygon tubing, or equivalent
- Small vacuum pump for filtration
- Glass petri dishes
- Jaffe washer
- Carbon evaporator rods
- Wash bottles, plastic
- Reagent alcohol

4.0 METHOD SUMMARY

Figure 1 presents a simplified overview of the TEM indirect preparation procedure for overloaded air samples and dust samples. As seen, there are two general indirect preparation procedures, one that includes ashing of the primary filter and one that does not include ashing of the primary filter.

Laboratory modification LB-000053 provides a list of which sample prefix codes shall be prepared using an ashing procedure and which should not be prepared using an ashing procedure. In cases where there is a conflict regarding sample type between the sample prefix as defined by the most recent version of LB-000053 and the chain of custody instructions, the chain of custody instructions take precedent. Additionally, once sample preparations have begun, there may be cases where the analyst determines that ashing is necessary to obtain acceptable filter loading. Samples for which ashing may be warranted include indoor air or dust samples collected from properties with elevated levels of organic particulates (e.g., due to cigarette smoke or use of a wood-burning stove). In these samples, ashing may further reduce particulate loading, thus allowing for an improved analytical sensitivity.

The sections below present the detailed steps associated with each procedure. For all indirect preparations, specimen preparation should be performed in a clean facility that is separate from both bulk and air preparation areas and preparation shall take place in a negative flow HEPA hood to prevent any possible contamination of the laboratory or personnel.

4.1 PROCEDURE 1: Indirect Preparation with Ashing

This procedure should be followed for air and dust samples where LB-000053 or the chain of custody form indicates that ashing should be performed. For the purpose of the Libby Superfund Site, air samples are defined as overloaded if there is >25% obscuration on the majority of the grid openings.

If there is no loose material present in the air cassette or adhering to the cowl, this procedure is generally similar to the indirect preparation method specified in ISO 13794, but has been modified to increase the total solution volume from 40 ml to 100 ml and to retain a portion of the original filter. The use of a 100 ml final volume is selected because it allows for preparation of a

series of indirect samples with volumes that are sufficiently large that secondary dilution is not needed to ensure uniform deposition on the filter.

If there is loose material present in the air cassette or adhering to the cowl, or if the sample is a dust sample, a portion of the original filter is not retained for archive, since it is assumed that there will be uneven loading on the filter. Because of this, an archived portion of the original filter is unlikely to be representative. In this case, the indirect preparation procedure is similar to the method specified in ASTM D-5755, but has been modified to include an ashing of the primary filter.

4.1.1 Carefully wet-wipe the exterior of the cassettes to remove any possible contamination prior to taking the cassettes into the clean preparation area.

4.1.2 Within a safety hood, carefully open the cassette and verify if there is any loose material in the cassette or adhering to the cowl. **If this is an air sample and there is no visible loose material present, proceed to Step 4.1.6.**

4.1.3 Any loose material that is present in the cassette should be poured into a disposable 50 ml glass beaker or similar container.

4.1.4 Using freshly cleaned forceps, remove the sample collection filter from the sampling cassette and place it in the same disposable 50 ml glass beaker or similar container with the side containing the sample facing down.

4.1.5 Using a 50/50 alcohol/particle-free water solution, rinse any material adhering to the cowl into a new 25 mm diameter disposable filtration funnels. If the filtration unit does not come pre-assembled with the necessary components (e.g. contains a glass fiber filter instead of the required MCE filter), it will be necessary to disassemble the stock cassette as it comes from Whatman and discard the glass-fiber filter. Rinse the filter unit thoroughly with particle free water and reassemble the filter unit using a cellulose support pad (Pall 66238), a 5.0µm pore size MCE diffuser filter (Enviropore FILA500A025A), and a 0.2 µm pore size MCE final filter (Enviropore FILA020A025A). Apply vacuum. When all solution has passed through, rinse sides of filter funnel with a stream of particle free water to dislodge any particulate that might be adhering to the sides of the filter funnel. Once filtration is complete turn off vacuum, remove filter from unit and dry. Once the filter is dry, place it in the container with the original filter and **proceed to Step 4.1.8**.

4.1.6 Using freshly cleaned forceps, remove the sample collection filter from the sampling cassette and place it on a clean glass microscope slide that will be used as a cutting surface. Using a freshly cleaned curved scalpel blade, cut off ½ of the filter (estimate the ½ as precisely as possible as this affects the final concentration) with a rocking motion.

4.1.7 Place the remaining portion of the original filter in archive. (Note: In cases where an initial direct preparation of an air sample was attempted and found to be overloaded, this archive portion will be approximately ¼ of the original filter.) Place ½ of the primary filter

in a clean, single use disposable glass container with the side containing the sample facing down.

4.1.8 Cover the container with aluminum foil, forming a tight seal around the mouth.

4.1.9 Perforate the foil in 15-20 places with a syringe needle to allow for gas exchange during plasma ashing.

4.1.10 Place the sample container in the plasma asher chamber. Depending on the size of the plasma asher chamber, several samples may be ashed simultaneously.

4.1.11 Operate the plasma asher using the minimum power at which a glow-discharge is observed, until the filter appears to be completely ashed. Loss of particulate and fibers from the container will occur if the plasma asher is operated at excessive radio-frequency power. During ashing of mixed cellulose ester (MCE) filters, a critical point is reached during the oxidation at which a sudden, violent ignition may occur if the radio-frequency power is excessive. This may result in a loss of fibers from the container, contamination of the interior of the chamber, and possible cross-contamination of the samples. For this reason, ashing of the blank should be observed closely during the early stages of oxidation, in order to ensure that the radio-frequency power setting is such that sudden ignition does not occur.

4.1.12 After 100% ashing is complete based on visual observation, increase the plasma asher power to maximum and ash for a period of one additional hour.

4.1.13
While final ashing is in progress, set up the filtration system to be used. In order to minimize the chances of contamination, only 25 mm diameter disposable filtration funnels shall be used. If the filtration unit does not come pre-assembled with the necessary components (e.g. contains a glass fiber filter instead of the required MCE filter), it will be necessary to disassemble the stock cassette as it comes from Whatman and discard the glass-fiber filter. Rinse the filter unit thoroughly with particle free water and reassemble the filter unit using a cellulose support pad (Pall 66238), a 5.0µm pore size MCE diffuser filter (Enviropore FILA500A025A), and a 0.2 µm pore size MCE final filter (Enviropore FILA020A025A). Filter as usual, using restraint with the amount of vacuum applied to avoid uneven loading. Add 20 ml of particle-free water to the filtration apparatus prior to applying vacuum and introduction of the sample suspension. When seating the filters in the filtration unit, it is essential that the vacuum be evenly applied to help ensure an even distribution of particulate on the filter. There should be no air bubbles or surface abnormalities anywhere in the filter assemblage. This is accomplished through wetting each successive filter as it is placed in the filtration unit and applying a light vacuum. This will ensure that the filters are flat and that there are no air bubbles.

4.1.14 After ashing is complete, admit air slowly to the chamber and remove the samples from the plasma asher chamber and place back into a safety hood.

4.1.15 Remove the aluminum foil from the top of the sample container.

4.1.16 Using particle free water in a squirt bottle, carefully rinse the ashed residue from the ashing container into a clean disposable sample container of at least 100 ml with a watertight lid, such as a sealed specimen cup. Rinse the residue into the 100 ml container to an initial volume of approximately 90 ml. Adjust pH to approximately 3-4 using a 10% solution of glacial acetic acid, and checking with pH paper. Bring the final volume to 100 ml and cap tightly.

4.1.17 Briefly hand shake (3 seconds) the capped container containing the sample suspension.

4.1.18 Place the container in a calibrated tabletop ultrasonic bath and sonicate at 50 - 100 nW/ml for three minutes. The liquid level in the bath should be ½ to ¾ the height of the sample containers. Wipe the outside of the sample containers dry when removing them from the bath.

4.1.19 After sonication, lightly hand shake the suspension for 3 seconds, and allow it to stand undisturbed for 2 minutes to allow large particles to settle to the bottom or float to the top.

4.1.20 For each sample, prepare three secondary filters by applying volumes of 50 ml, 25 ml, and 10 ml. For air samples where the direct preparation proves to be overloaded, it is acceptable to filter aliquot volumes other than the usual 10 ml, 25 ml, and 50 ml series, either a greater or lesser volume, in order to produce a sample with the highest possible f-factor without violating the overload criteria. Draw each aliquot to be filtered with the same pipette and dispense into the appropriate filter funnel. Avoid pipetting any large settled or floating particles. Apply vacuum to the filtration apparatus to draw each volume through the filter. For samples where the 10 ml aliquot filter is obviously overloaded and a secondary dilution will be required (see 4.1.21), it is not necessary to attempt to filter the 25 ml and 50 ml aliquots through 25 mm filter units.

4.1.21 If a preliminary observation of the 10 ml secondary filter appears overloaded take 10 ml of the remaining volume and dilute to 100 ml. From this secondary dilution, prepare a second series of filters using 50 ml, 25 ml, and 10 ml (corresponding to 5 ml, 2.5 ml, and 1 ml of the original suspension). Based on the original 10 ml aliquot filter loading, it is acceptable to filter aliquot volumes other than the usual 10 ml, 25 ml, and 50 ml series in order to produce a sample with the highest possible f-factor without violating the overload criteria. In some instances, it may be necessary to perform a tertiary serial dilution, taking 10 ml of the secondary dilution, adding it to 90 ml of particle free water, and filtering another series of aliquots of 10 ml, 25 ml, and 50 ml.

4.1.22 Disassemble the filtration units. Carefully remove the filters from the filtration apparatus using fine forceps, being careful to only touch the inactive rim of the filter that has not been exposed to the sample. Place each filter in a labeled petri dish or other similar container, active side up and dry.

4.1.23 Select the secondary filter from the dilution series yielding the largest possible f-factor (highest possible volume) which does not violate the criteria for an overloaded sample. Experience has shown that a light staining of the filter will yield a suitable preparation for analysis.

4.1.24 Perform a standard TEM sample preparation procedure.

4.1.25 If TEM examination of the lowest volume aliquot filtered is deemed overloaded (>25% particulate), consult with the Libby laboratory coordinator (CDM) to select the most appropriate next step.

4.1.26 Carefully label and place each of the unused secondary filters and the remaining portion of the selected secondary filter in archive.

4.1.27 Place any remaining sample solution in a graduated cylinder or pipet. The largest known quantity of the remaining solution should be filtered through a 25 mm disposable filtration unit with a $\leq$ 0.22 µm/5.0 µm pore size MCE filter set in conjunction with a cellulose support pad and dried after removal from the filtration unit. A larger diameter (e.g. 47 mm) filtration unit with the same filter configuration may be used as needed to avoid situations where a 25 mm diameter filter may become obstructed with material. The dried filter shall be placed in an appropriate container, and labeled with the sample number, filter type, and volume applied to the filter. This filter will then be archived with the other archived filters from the sample.

4.1.28 Discard the remaining portion of the sample solution using standard laboratory protocols.

4.2 PROCEDURE 2: Indirect Preparation without Ashing

This procedure should be followed for air and dust samples where LB-000053 or the chain of custody form indicates that ashing should not be performed. For the purpose of the Libby Superfund Site, samples are defined as overloaded if there is >25% obscuration on the majority of the grid openings.

If there is no loose material present in the air cassette or adhering to the cowl, this procedure is generally similar to the indirect preparation method specified in ASTM D-5755, but has been modified to allow for an archive of the original filter.

If there is loose material present in the air cassette or adhering to the cowl, or if the sample is a dust sample, a portion of the original filter is not retained for archive, since it is assumed that there will be uneven loading on the filter. Because of this, an archived portion of the original filter is unlikely to be representative. In this case, the indirect preparation procedure is equivalent to the method specified in ASTM D-5755.

4.2.1 Carefully wet-wipe the exterior of the cassettes to remove any possible contamination prior to taking the cassettes into the clean preparation area.

4.2.2 Carefully open the cassette and verify if there is any loose material in the cassette or adhering to the cowl. **If this is an air sample and there is no visible loose material present, proceed to Step 4.2.5**.

4.2.3 Using a 50/50 alcohol/particle-free water solution, rinse any material adhering to the cowl down onto the sample collection filter (still inside the sampling cassette).

4.2.4 Using freshly cleaned forceps, remove the sample collection filter from the sampling cassette and place it into a clean disposable sample container of at least 100 ml with a watertight lid, such as a sealed specimen cup. **Proceed to Step 4.2.7**.

4.2.5 Using freshly cleaned forceps, remove the sample collection filter from the sampling cassette and place it on a clean glass microscope slide that will be used as a cutting surface. Using a freshly cleaned curved scalpel blade, cut off ½ of the filter (estimate the ½ as precisely as possible as this affects the final concentration) with a rocking motion.

4.2.6 Place the remaining portion of the original filter in archive. (Note: In cases where an initial direct preparation of an air sample was attempted and found to be overloaded, this archive portion will be approximately ¼ of the original filter.) Place ½ of the primary filter in a clean disposable sample container of at least 100 ml with a watertight lid, such as a sealed specimen cup.

4.2.7 Bring the total volume of the suspension up to approximately 90 ml using particle-free water only.

4.2.8 Adjust the suspension to a pH of 3-4 using a 10 % solution of acetic acid. Use pH paper to test.

4.2.9 Bring the total volume up to 100 ml using particle-free water and cap tightly.

4.2.10 Set up the filtration system to be used. In order to minimize the chances of contamination, only 25 mm disposable filtration funnels (such as Whatman cat. #:1922-1820) shall be used. If the filtration unit does not come pre-assembled with the necessary components (e.g. contains a glass fiber filter instead of the required MCE filter), it will be necessary to disassemble the stock cassette as it comes from Whatman and discard the glass-fiber filter. Rinse the filter unit thoroughly with particle free water and reassemble the filter unit using a cellulose support pad (Pall 66238), a 5.0µm pore size MCE diffuser filter (Enviropore FILA500A025A), and a 0.2 µm pore size MCE final filter (Enviropore FILA020A025A). Filter as usual, using restraint with the amount of vacuum applied to avoid uneven loading. Add 20 ml of particle-free water to the filtration apparatus, prior to applying vacuum and introduction of the sample suspension. When seating the filters in the filtration unit, it is essential that the vacuum be evenly applied resulting in even distribution. There should be no air bubbles or surface abnormalities anywhere in the filter assemblage. This is accomplished through wetting each successive filter as it is placed in the filtration unit and

applying a light vacuum. This will assure that the filters are flat and that there are no air bubbles. Ensure that suspension is filtered within 24 hours to avoid problems associated with bacterial and fungal growth.

4.2.11 Briefly hand shake (3 seconds) the capped container containing the sample suspension.

4.2.12 Place the container in a calibrated tabletop ultrasonic bath and sonicate at 50 - 100 nW/ml for three minutes.

4.2.13 After sonication, lightly hand shake the suspension for 3 seconds, and allow it to stand undisturbed for 2 minutes to allow large particles to settle to the bottom or float to the top.

4.2.14 For each sample, prepare three secondary filters by drawing aliquots of 50 ml, 25 ml, and 10 ml. For air samples where the direct preparation is overloaded, it is acceptable to filter aliquot volumes other than the usual 10 ml, 25 ml, and 50 ml series (either greater or lesser volumes) in order to produce a sample with the highest possible f-factor without violating the overload criteria. Draw each aliquot to be filtered with the same pipette and dispense into the appropriate filter funnel. Avoid pipetting any large settled or floating particles. Apply vacuum to the filtration apparatus to draw each volume through the filter. For samples where the 10 ml aliquot filter is obviously overloaded and a secondary dilution will be required (see 4.2.15), it is not necessary to attempt to filter the 25 ml and 50 ml aliquots through 25 mm filter units.

4.2.15 If a preliminary observation of the 10 ml secondary filter appears overloaded take 10 ml of the remaining volume and dilute to 100 ml. From this secondary dilution, prepare a second series of filters using 50 ml, 25 ml, and 10 ml (corresponding to 5 ml, 2.5 ml, and 1 ml of the original suspension). Based on the original 10 ml aliquot filter loading, it is acceptable to filter aliquot volumes other than the usual 10 ml, 25 ml, and 50 ml series (either greater or lesser volumes) in order to produce a sample with the highest possible f-factor without violating the overload criteria. In some instances, it may be necessary to perform a tertiary serial dilution, taking 10 ml of the secondary dilution, adding it to 90 ml of particle free water, and filtering another series of aliquots of 10 ml, 25 ml, and 50 ml.

4.2.16 Disassemble the filtration unit. Carefully remove the filter from the filtration apparatus using fine forceps, being careful to only touch the inactive rim of the filter that has not been exposed to the sample. Place the filter in a labeled petri dish or other similar container, active side up and dry.

4.2.17 Select the secondary filter from the dilution yielding the largest possible f-factor (highest volume) which does not violate the criteria for an overloaded sample. Experience has shown that a light staining of the filter will yield a suitable preparation for analysis.

4.2.18 Perform a standard TEM sample preparation procedure.

4.2.19 If TEM examination of the lowest volume aliquot filtered is deemed overloaded, consult with the Libby laboratory coordinator (CDM) to select the most appropriate next step.

4.2.20 Place each of the unused secondary filters and the remaining portion of the selected secondary filter in archive.

4.2.21 Place any remaining sample solution in a graduated cylinder or pipet and add to a prepared 25 mm filtration unit containing a $\leq$ 0.22 µm/5.0 µm pore size filter set with a cellulose support pad in a disposable filtration unit with a small volume of particle free water to facilitate the production of a homogeneous solution and record the volume of sample solution added. A larger diameter (e.g. 47 mm) filtration unit with the same filter configuration may be used as needed to avoid situations where a 25 mm diameter filter may become obstructed with material. Add 10 ml particle free water to the sample container containing the residual filter and sonicate for three minutes. Add this solution to the filtration unit for the corresponding filtration unit for each sample as described in the first part of this paragraph. Do not include this 10 ml in the volume calculation of the sample solution added. This solution should then be filtered through the filtration unit and dried after removal from the filtration unit. The dried filter shall be placed an appropriate container, and labeled with the sample number, filter type, and volume applied to the filter. This filter will then be archived with the other archived filters from the sample.

4.2.22 Discard the remaining portion of the sample solution using standard laboratory protocols.

5.0 DOCUMENTATION AND ARCHIVE STORAGE

Project-specific Index IDs are recorded on all air samples. During each indirect preparation step, this Index ID is noted on the sample-specific beakers, containers, and filtration units.

In those cases where no loose material is present in the cassette or adhering to the cowl, the remaining portion of the original primary filter is placed in a suitable container and clearly labeled with the sample number and indicated that it is the original primary filter. In those cases where secondary or tertiary filters are prepared, all filters or remnants of filters will be archived into suitable containers, and clearly labeled with the sample number and the volume of the aliquot applied to each filter.

Analysis-specific details about the indirect preparation will be recorded in the sample TEM electronic data deliverable (EDD) spreadsheet. In the TEM EDD, if the sample is prepared using Procedure 1 (see Section 4.1) the preparation method should be identified as “IA – Indirect, ashed” and the appropriate inputs should be recorded in the fields provided. If the sample is prepared using Procedure 2 (see Section 4.2), the preparation method should be identified as “I – Indirect” and the appropriate inputs should be recorded in the fields provided. The spreadsheet is designed to automatically calculate the dilution factor, or f-factor, which is used in the calculation the sample air or dust concentration.

6.0 QUALITY ASSURANCE

All quality control sample results will be monitored for potential contamination. If sample results indicate cross-contamination, the laboratory will identify the affected samples and notify the USEPA Regional Chemist and project laboratory coordinator (CDM). Laboratory procedures will be re-assessed and appropriate changes will be made and documented accordingly by the project laboratory coordinator.

6.1 Lot Blanks

All cassettes utilized in the Libby project are screened for contamination by either TEM analysis or a combination of TEM and PCM analysis. One lot blank is prepared and analyzed from each carton of cassettes prior to using the lot of cassettes for sampling. The entire carton of cassettes will be rejected if any asbestos fiber is detected on the lot blank.

6.2 Filter blanks

Prior to filtration of the sample aliquot, 100ml particle-free water should be filtered. Acceptance criteria for filter blanks are as specified for laboratory blanks in the latest version of laboratory modification of LB-000029.

6.3 Plasma asher blanks

To ensure that contamination is not introduced during the ashing process, a container with an unused filter should be run as a blank with each batch of samples ashed. This sample will be prepared using the standard TEM sample preparation procedure and examined as per the established QC sequence. Acceptance criteria for plasma asher blanks are as specified for laboratory blanks in the latest version of laboratory modification of LB-000029.

7.0 DECONTAMINATION

All non-disposable equipment used during sample preparation must be decontaminated prior to use. Because the prescribed filtration units used to prepare the secondary filters are disposable, decontamination of filtration units is not required.

8.0 GLOSSARY

EDD - Electronic Data Deliverable. A Libby-specific spreadsheet designed to capture the detailed analysis and raw structure data generated during a TEM analysis. Contact the project laboratory coordinator (CDM) for the current TEM spreadsheet version.

HEPA - High Efficiency Particulate Air

MCE - Mixed Cellulose Ester

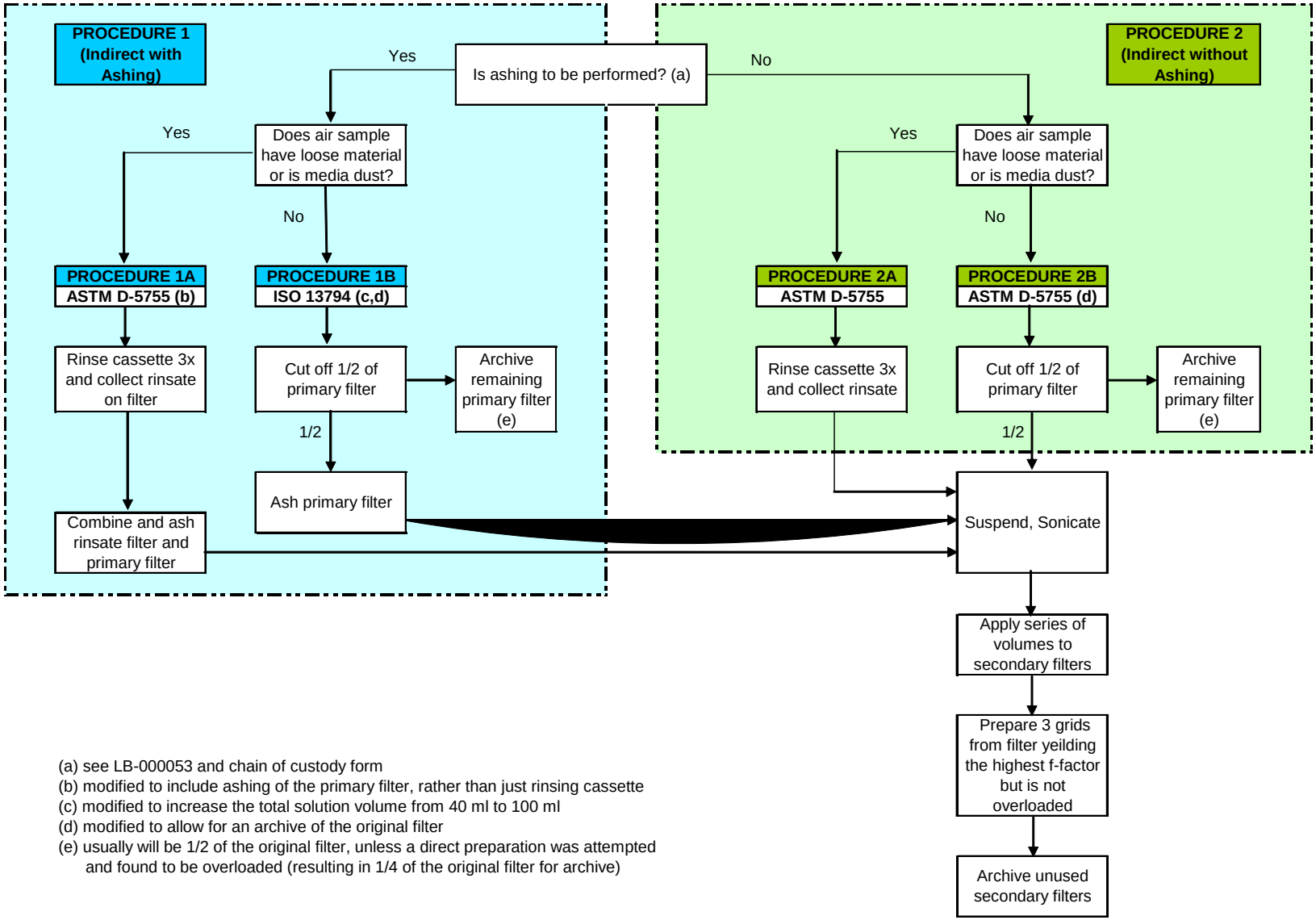
TEM - Transmission Electron Microscopy

9.0 REFERENCES

ISO 13794. Ambient air - Determination of asbestos fibres - Indirect-transfer transmission electron microscopy method. International Organization for Standardization (ISO) 13794:1999. November 15, 1999.

ASTM D-5755. Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading. ASTM D 5755-03. October 2003.

FIGURE 1. INDIRECT PREPARATION OF OVERLOADED AIR SAMPLES AND DUST SAMPLES FOR TEM ANALYSIS



(a) see LB-000053 and chain of custody form
(b) modified to include ashing of the primary filter, rather than just rinsing cassette
(c) modified to increase the total solution volume from 40 ml to 100 ml
(d) modified to allow for an archive of the original filter
(e) usually will be 1/2 of the original filter, unless a direct preparation was attempted and found to be overloaded (resulting in 1/4 of the original filter for archive)

Date: September 6, 2011 OU3 SOP 8 (Rev. 2)

Title: SAMPLE HANDLING AND SHIPPING

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager		9/14/11
SOP Author		9/15/11

Revision Number	Date	Reason for Revision
0	09/26/2007	--
1	04/04/2011	Remove shipping peanuts as a viable cushioning material for the shipment of samples
2	09/06/2011	Add details of shipping methods for asbestos filters

1.0 INTRODUCTION

This standard operating procedure (SOP) is based on MWH SOP-09, Sample Handling and Shipping, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3. This SOP describes the requirements for sample handling, storage and shipping. The purpose of this SOP is to define sample management activities as performed from the time of sample collection to the time they are received by the laboratory.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Chain-of-Custody: An accurate written record of the possession of each sample from the time of collection in the field to the time the sample is received by the designated analytical laboratory.

Sample: Physical evidence collected for environmental measuring and monitoring.
For the purposes of this SOP, sample is restricted to solid, aqueous, air, or waste matrices. This SOP does not cover samples collected for lithologic description nor does it include remote sensing imagery or photographs (refer to SOP-9 for field documentation procedures).

Sampler: The individual who collects environmental samples during fieldwork.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, and etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: The Project Manager is responsible for ensuring that the requirements for sample management are included in the appropriate project plans. The Project Manager is

responsible for coordinating sample management efforts with input from other key project staff and applicable government agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader and/or Field Hydrogeologist, Geologist or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Field Technician: Responsible for sample collection, documentation, packaging, and shipping. Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks.

5.0 PROCEDURES

5.1 Applicability

The information in this SOP may be used by direct reference or incorporated into project-specific plans. Deviations or modifications to procedures addressed herein must be brought to the attention of, and approved by, applicable government agencies.

5.2 Sample Management

Sample Containers: The sample containers to be used will be dependent on the sample matrix and analyses desired, and are specified in the project FSP. Only certified pre-cleaned sample containers will be used. Sample containers will be filled with adequate headspace (approximately 10 percent) for safe handling upon opening, except containers for volatile organic compound (VOC) analyses, which will be filled completely with no headspace. This no-headspace requirement applies to both soil and groundwater samples.

Once opened, the containers will be used immediately. If the container is used for any reason in the field (e.g., screening) and not sent to the laboratory for analysis, it will be discarded. Prior to discarding the contents of the used container and the container, disposal requirements will be evaluated. When storing before and after sampling, the containers will remain separate from solvents and other volatile organic materials. Sample containers with preservatives added by the laboratory will not be used if held for an extended period on the job site or exposed to extreme heat conditions. Containers will be kept in a cool, dry place. For preserved samples (except VOCs), the pH of the sample will be checked following collection of the sample. If the pH is not at the required level, additional preservative (provided by the laboratory) will be added to the sample container.

Numbering and Labeling: Refer to OU3 SOP-9.

Custody Seals. Custody seals with the date and initials of the sampler will be used on each shipping container to ensure custody. The custody seal will be placed on opposites sides of the cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of the liner bag inside of the cooler.

Chain-of-Custody: COC procedures require a written record of the possession of individual samples from the time of collection through laboratory analyses. A sample is considered to be in custody if it is:

- In a person's possession
- In view after being in physical possession
- In a secured condition after having been in physical custody
- In a designated secure area, restricted to authorized personnel

The COC record will be used to document the samples taken and the analyses requested. Refer to SOP-9 Attachment 2 for the OU3-specific COC form. Information recorded by field personnel on the COC record will include the following:

- Sample identifier (Index ID)
- Date and time of collection
- Sample matrix
- Preservation
- Type of analyses requested
- Unique COC number
- Lab being shipped to
- Signature of individuals involved in custody transfer (including date and time of transfer)
- Airbill number (if appropriate)
- Any comments regarding individual samples (e.g., organic vapor meter readings, special instructions).

COC records will be placed in a waterproof plastic bag (e.g., Ziploc®), taped to the inside lid of the cooler or placed at the top of the cooler, and transported with the samples. Signed airbills will serve as evidence of custody transfer between the field sampler and courier, as well as between the courier and laboratory. If a carrier service is used to ship the samples (e.g., Federal Express, etc.), custody will remain with the courier until it is relinquished to the laboratory. Upon receiving the sample cooler, a laboratory representative should sign in the receiving box of the COC, thus establishing custody. The sampler will retain copies of the COC record and airbill.

Sample Preservation/Storage: The requirements for sample preservation are dependent on the desired analyses and the sample matrix, and are specified in the FSP.

5.3 Sample Shipping

The methods and procedures described in this SOP were developed from these sources:

- 49 CFR 173. Shippers – Shippers – General Requirements for Shipping. United States Code of Federal Regulations available online at http://www.gpoaccess.gov/cfr/index.html

- 49 CFR 178. Specifications for Packaging. United States Code of Federal Regulations available online at http://www.gpoaccess.gov/cfr/index.html

- ASTM D 4220. Standard Practice for Preserving and Transporting Soil Samples. American Society for Testing and Materials available online at http://www.astm.org/

- ASTM D 4840. Standard Practice for Sampling Chain-of-Custody Procedures. American Society for Testing and Materials available online at http://www.astm.org/

Procedures for packaging and transporting samples to the laboratory are dependent on the chemical, physical, and hazard properties of the material. The procedures may also be based on an estimation of contaminant concentrations/properties in the samples to be shipped. Samples will be identified as environmental samples, excepted quantities samples, limited quantities samples, or standard hazardous materials. Environmental samples are defined as solid or liquid samples collected for chemical or geotechnical analysis. Excepted quantities involve the shipment of a few milliliters of either an acid or base preservative in an otherwise empty sample container. Limited quantities are restricted amounts of hazardous materials that may be shipped in generic, sturdy containers. Standard hazardous material shipments require the use of stamped/certified containers. All samples will be packaged and shipped or hand delivered to the laboratories the same day of sample collection, unless otherwise specified in the project-specific FSPs.

The following paragraphs describe standard shipping procedures for different types of samples. Any exceptions to these procedures will be defined in the FSP. It is the responsibility of the sampler to refer to the U.S. Department of Transportation (DOT) (http://hazmat.dot.gov/regs/rules.htm) regulations when dealing with a substance not addressed in this SOP for requirements and limitations associated with the shipment.

Sample Shipping via Commercial Carrier:

*Aqueous or Solid Samples***:** Samples will be packaged and shipped to the laboratories the same day of sample collection, unless otherwise specified in the FSP and depending on holding time requirements for individual samples. For aqueous or solid samples that are shipped to the laboratory via a commercial carrier the following procedures apply:

- Sample labels will be completed and attached to sample containers.
- The samples will be placed upright in a waterproof metal (or equivalent strength plastic) ice chest or cooler.
- For shipments containing samples for volatile organic analysis, include a trip blank.
- Ice in double Ziploc® bags (to prevent leakage) will be placed around, among, and on top of the sample bottles. Enough ice will be used so that the samples will be chilled and maintained at 4°C ± 2°C during transport to the laboratory. Dry ice or blue ice will not be used.
- To prevent the sample containers from shifting inside the cooler, the remaining space in the cooler will be filled with inert cushioning material, such as additional bubble pack or cardboard dividers, such that the sample containers remain upright and do not break.
- Tape shut the cooler's drain plug
- The original copy of the completed COC form will be placed in a waterproof plastic bag and taped to the inside of the cooler lid or placed at the top of the cooler.
- The lid will be secured by wrapping strapping tape completely around the cooler in two locations.
- Mark the cooler with arrow labels indicating the proper upright position of the cooler.
- Custody seals consisting of security tape with the date and initials of the sampler will be used on each shipping container to ensure custody. Two signed custody seals will be placed on the cooler, one on the front and one on the back.
- A copy of the COC record and the signed air bill will be retained for the project files.
- Affix a label containing the name and address of the shipper to the outside of the cooler

Hand-Delivered Samples: For aqueous or solid samples that will be hand carried to the

laboratory, the same procedures apply.

Excepted Quantities: Usually, corrosive preservatives (e.g., hydrochloric acid, sulfuric acid, nitric acid, or sodium hydroxide) are added to otherwise empty sample bottles by the analytical laboratory prior to shipment to field sites. However, if there is an occasion whereby personnel are required to ship bottles with these undiluted acids or bases, the containers will be shipped in the following manner:

1. Each individual sample container will have not more than 30 milliliters of preservative.

2. Collectively, the preservative in these individual containers will not exceed a volume of 500 milliliters in the same outer box or package.
3. Despite the small quantities, only chemically compatible material may be placed in the same outer box, (e.g., sodium hydroxide, a base, must be packaged separately from the acids).

4. Federal Express will transport nitric acid only in concentrations of 40 percent or less.

5. A "Dangerous Goods in Excepted Quantities" label will be affixed to the outside of the outer box or container. Information required on the label includes:

    - Signature of Shipper
    - Title of Shipper
    - Date
    - Name and Address of Shipper
    - Check of Applicable Hazard Class
    - Listing of UN Numbers for Materials in Hazard Classes

Limited Quantities: Occasionally, it may become necessary to ship known hazardous materials, such as pure or floating product. DOT regulations permit the shipment of many hazardous materials in "sturdy" packages, such as an ice chest or cardboard box (not a specially constructed and certified container), provided the following conditions are met:

1. Each sample bottle is placed in a plastic bag, and the bag is sealed. Each VOC vial will be placed in a sealable bag. As much air as possible is squeezed from the bag before sealing. Bags may be sealed with evidence tape for additional security.

2. Or each bottle is placed in a separate paint can, the paint can is filled with vermiculite, and the lid is affixed to the can. The lid must be sealed with metal clips, filament, or evidence tape. If clips are used, the manufacturer typically recommends six clips.

3. The cans are placed upright in a cooler that has had the drain plug taped shut inside and outside, and the cooler is lined with a large plastic bag. Approximately 1 inch of adsorbent material sufficient to retain any liquid that may be spilled, is placed in the bottom of the liner. Only containers having chemically compatible material may be packaged in each cooler or other outer container.

4. The COC record is sealed inside a plastic bag and placed inside the cooler. The sampler retains one copy of the COC record. The laboratory will be notified if the sample is suspected of containing any substance for which the laboratory personnel should take safety precautions.

5. The cooler is shut and sealed with strapping tape (filament type) around both ends. Two signed custody seals will be placed on the cooler, one on the front and one on the back. Additional seals may be used if the sampler and/or shipper consider more seals to be necessary. Wide, clear tape will be placed over the seals to ensure against accidental breakage.

6. The following markings are placed on the side of the cooler:
    - Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA Dangerous Goods Regulations [DGR])
    - UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
    - Shipper's name and address
    - Consignee's name and address
    - The words "LIMITED QUANTITY"
    - Hazard Labels (Column E, List of Dangerous Goods, Section 4, IATA DGR)
    - Two Orientation (Arrow) labels placed on opposite sides.

7. The Airbill/Declaration of Dangerous Goods form is completed as follows:
    - Shipper's name and address
    - Consignee's name and address
    - Services, Delivery & Special Handling Instructions
    - Cross out "Cargo Aircraft Only" in the Transport Details Box
    - Cross out "Radioactive" under Shipment Type
    - Nature and Quantity of Dangerous Goods
        - Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA DGR)
        - Class or Division (Column C, List of Dangerous Goods, Section 4, IATA DGR)

- UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
- Packing Group (Column F, List of Dangerous Goods, Section 4, IATA DGR)
- Subsidiary Risk, if any (Column D, List of Dangerous Goods, Section 4, IATA DGR)
- Quantity and type of packing (number and type of containers: for example, "3 plastic boxes", and the quantity per container, "2 L", is noted as "3 Plastic boxes X 2 L" This refers to 3 plastic boxes (coolers are referred to as plastic boxes) with 2 liters in each box.
- Packing Instructions (Column G, List of Dangerous Goods, Section 4, IATA DGR).
- Note: Only those Packing Instructions in Column G that begin with the letter "Y" may be used. These refer specifically to the Limited Quantity provisions.
- Authorization (Write in the words Limited Quantity)
- Emergency Telephone Number (List 800-535-5053. This is the number for INFOTRAC.)
- Printed Name and Title, Place and Date, Signature.

Shipping Filters for Asbestos Analysis: Protocols for shipment of filters that have been collected for asbestos analysis are presented below.

Shipment of Filters in Cassettes: Filter cassettes that have been used to collect samples of asbestos for microscopic analysis shall be stored and shipped in the boxes the cassettes were originally supplied in. The cassettes shall not be stored in plastic bags, since this may lead to electrostatic change that could disrupt the filter loading. The cassettes will be placed in the original box with the inflow port of the cowl facing up, with plugs on both ends. Each cassette must be clearly labeled with the sample identifier. Cassettes will be shipped to the analytical laboratory in a cooler with a handle on top. The handle will help prevent the cooler from being shipped upside down and will help ensure the cassettes remain vertical with the open end of the cowl facing up.

Shipment of Isolated Filters Intended for Indirect Preparation: Filters that have been collected and are planned for indirect analysis shall be placed in 20-mL glass scintillation vials for shipment. Each vial shall contain one filter, and the sample identifier shall be clearly marked on the vial. The vial shall be capped before shipment. Shipment shall be in a box with dividers that maintain the vials in an upright position and prevent the vials from touching.

Shipment of Isolated Filters Intended for Direct Preparation: Shipment of filters intended for direct preparation is not generally recommended due to the potential for fiber loss from the filter during shipment. However, when shipment is necessary, each filter will be placed in a small

glass Petri dish and attached to the bottom (filter side up) with two pieces of tape. The tape must be carefully applied so that only the outer rim of the filter is contacted by the tape. The top of the Petri dish shall then be attached with tape, and the sample identifier shall be clearly marked on the dish. When multiple samples are to be shipped, the Petri dishes, each containing one sample, shall be placed in verticals stacks of up to 10 dishes each, and the dishes in each stack shall be attached to each other with tape.

Standard Hazardous Materials: Shipment of standard hazardous materials presents the most difficulty and expense. However, there may be occasion whereby a hazardous material cannot be shipped under the Limited Quantity provisions, (e.g., where there is no Packing Instruction in Column G, List of Dangerous Goods, IATA Dangerous Goods Regulations, that is preceded by the letter "Y").

In such cases, the general instructions noted above but for non-Limited Quantity materials will apply, with one important difference: standard hazardous materials shipment requires the use of certified outer shipping containers. These containers have undergone rigid testing and are, therefore, designated by a "UN" stamp on the outside, usually along the bottom of a container's side. The UN stamp is also accompanied by codes specifying container type, packing group rating, gross mass, density, test pressure, year of manufacturer, state of manufacturer, and manufacturer code name. The transport of lithium batteries in Hermit Data Loggers is an example of a standard hazardous material where only a designated outer shipping container may be used.

5.4 Holding Times

The holding times for samples will depend on the analysis and the sample matrix. Refer to the FSP for holding times requirements.

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

All sample shipments must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that handling and shipment documentation has been completed per this procedure and other procedures referenced herein.

7.0 DECONTAMINATION

All shipment coolers shall be maintained clean of sampled material to avoid exposure during shipment. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

8.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.
Enforcement Considerations for Evaluations of Uncontrolled Hazardous Waste Disposal Sites by Contractors, Draft, Appendix D, April 1980.

APPENDIX B

LIBBY LABORATORY MODIFICATIONS FOR TEM ANALYSES

LB-000018
LB-000019
LB-000028
LB-000029b
LB-000030
LB-000066c
LB-000085

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Request for Modification
To
Laboratory Activities
LB-000016

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, [TEM-ISO 10312], PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other:_____

Requester: Jeanne Orr Title: President
Company: Reservoirs Environmental, Inc. Date: December 2, 2002

Description of Modification:
Permanent modifications and clarifications to the Transmission Electron Microscopy analysis of air samples using ISO 10312. The purpose of the attached is to document permanent historic modifications & clarifications.

Reason for Modification:
To optimize the efficiency of air sample analysis and to provide consistency in analytical procedures and data recording in the project laboratories.

Potential Implications of this Modification:
Modifications reflect changes necessary to clarify ISO requirements in relation to project-specific issues. No negative implications to these modifications are anticipated. Positive implications are consistency in procedures between and within project laboratories and documentation of those procedures.

Laboratory Applicability (circle one): [All] Individual(s) _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

[Permanent] (complete Proposed Modification Section) Effective Date: HISTORIC

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by TEM analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):
Please see the attached for the description of the TEM-ISO clarifications/modifications

Technical Review: R.K. Mahoney EMSL Date: 23 April 2003
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 4 April 2003
(Volpe: Mark Raney)

Approved By: _____ Title: Project Chemist Date: 3 April 2003
(USEPA: Mary Goldade)

1. **Modification:**
   The ISO method requirement is if the specimen grid exhibits more than approximately 10% obscuration on the majority of the grid openings, the specimen shall be designated as overloaded. A rejection criteria of >25% obscuration and <50% intact grid openings will be used for this project. The 25 % overload criteria resulted from various communications that took place 29 December 1999 between EPA Region 8, Camp Dresser McKee, Volpe Center, and Reservoirs.
2. **Modification:**

   ISO 10312 is a direct preparation method. If samples are visibly overloaded or contain loose debris and they have not been previously analyzed (the filter is whole) they will be prepared indirectly according to procedures described in ASTM D5755-95. If the sample has been previously analyzed or rejected in the microscope (section removed from the filter), prepare the sample indirectly according to EPA/540/2-90/005a by plasma ashing a portion of the original filter and depositing an aliquot on a secondary filter. Secondary filters will be analyzed according to the ISO counting rules for this project. Calculations are modified to contain a dilution factor. This indirect preparation procedure is embraced to enable the capture of data from samples that otherwise would be rejected.

3. **Clarification:**
   Stopping rules for ISO analyses are completion of the grid opening on which the 100^{th} asbestos structure has been recorded, or a minimum of four grid openings. For this project, a maximum of ten grid openings will be read unless specifically instructed otherwise.

   If abundant chrysotile is present, the chrysotile count may be terminated at the end of the grid opening where the 100^{th} chrysotile structure is counted. The analysis will continue recording amphibole fibers only until the remaining grid openings to be analyzed are completed. The grid opening location designation will be followed by a "*" to indicate the grid openings where only amphibole asbestos was recorded, i.e. K6*.
   This clarification in structure counting and recording is to provide consistency in analytical procedures and data recording in the project laboratories.

4. **Modifications and clarifications: Structure counting and recording**
   a. **Modification:** Non-asbestos structures are not being recorded. This project-specific modification stems from our need only to quantify contaminants of concern: the asbestos levels at a given sample location.
   b. **Modification:** The overall dimensions of disperse clusters (CD) and disperse matrices (MD) will not be recorded in two perpendicular directions. The matrix type and individual structures associated with the matrix or cluster will be recorded as described in the ISO method.
   c. **Modification:** Structures that intersect a non-countable grid bar will be recorded on the count sheet but excluded from the structure density and concentration calculations.
   d. **Modification:** If a structure originates in one grid opening and extends into an adjacent grid opening, providing that it does not intersect a non-counting grid bar, the entire length of the fiber is recorded.
   e. **Clarification:** If a structure intersects both a countable and a non-countable grid bar, the observed length of the structure will be recorded.

   These modifications and clarifications in structure counting and recording are to provide consistency in analytical procedures and data recording in the project laboratories.

Mahoney, Ron

From: Raney, Mark [RANEY@VOLPE.DOT.GOV]
Sent: Tuesday, April 22, 2003 11:09 AM
To: 'Mahoney, Ron'
Subject: FW: VOLPE Approved MODS: LB-000015, LB-000016, and LB-000017


LB-000015_rev (MR 4-4-03 email...


LB-000016_rev (MR 4-4-03 email...


LB-000017_rev (MR 4-4-03 email...

FYI

> -----Original Message-----
> From: Raney, Mark
> Sent: Friday, April 04, 2003 9:31 AM
> To: 'Beckham, Richard'; 'Goldade.mary@EPAmail.epa.gov'; 'mgoldade@peakpeak.com'
> Cc: Autio, Anni
> Subject: VOLPE Approved MODS: LB-000015, LB-000016, and LB-000017
>
>
> Volpe provides approval to revised MODs LB-000015, LB-000016, & LB-000017 as attached. The attached MODs include the following changes to the previous versions (received 4/1/03).
>
> * The date indicated in the "Effective Date" field was removed and replaced with "HISTORIC"
> * Under the "Description of Modification" section the following sentence was added "The purpose of the attached is to document permanent historic modifications & clarifications."
>
> If you have any questions as to these changes or the reason behind them let me know. Please proceed with distribution of the accepted versions of the attached for final hardcopy signature.
>
> Mark.
>
> > <<LB-000015_rev (MR 4-4-03 email).doc>> > > <<LB-000016_rev (MR 4-4-03 email).doc>> > > <<LB-000017_rev (MR 4-4-03 email).doc>>
>
>
> -----Original Message-----
> From: Beckham, Richard [mailto:BeckhamRE@cdm.com]
> Sent: Tuesday, April 01, 2003 10:47 AM
> To: 'Goldade.mary@EPAmail.epa.gov'; 'RANEY@VOLPE.DOT.GOV';
> 'mgoldade@peakpeak.com'
> Cc: Autio, Anni
> Subject: FW: LB-000015, LB-000016, and LB-000017
>
>
> For your review and approval.
>
> - Richard Beckham
>
> -----Original Message-----
> From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]
> Sent: Monday, March 31, 2003 6:11 PM
> To: Beckham, Richard
> Subject: LB-000015, LB-000016, and LB-000017
>
>
> Richard,
>
> These should be final. The only recent revision is the addition of the
> Effective Date. These need to go to Mark and Mary for their final blessing.

> <<LB-000015(rev 3_31_03).doc>> <<LB-000016 rev. (3_31_03).doc>>
> <<LB-000017 rev(3_31_03).doc>>
>
> R.K. Mahoney
> Senior Analyst
> Special Projects Coordinator
> EMSL Analytical, Inc.
> Westmont, NJ
> 800.220.3675, x1218
> rmahoney@emsl.com
>
> << File: LB-000015(rev 3_31_03).doc >> << File: LB-000016 rev. (3_31_03).doc >> << File: LB-000017 rev(3_31_03).doc >>

Mahoney, Ron

From: Raney, Mark [RANEY@VOLPE.DOT.GOV]
Sent: Wednesday, April 23, 2003 9:02 AM
To: 'Mahoney, Ron'
Subject: FW: EPA APPROVED CONDITIONAL: LB-000015, LB-000016, and LB-000017


LB-000015(rev 3_31_03).doc


LB-000016 rev. (3_31_03).doc


LB-000017 rev(3_31_03).doc

Ron,

I almost forgot to forward you this....

See Mary's earlier email below, regarding EPA's approval for MODs LB-15, 16, & 17.

Let me know if you have any questions.

Mark.

-----Original Message-----
From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]
Sent: Thursday, April 03, 2003 5:49 PM
To: Beckham, Richard
Cc: Autio, Anni; 'mgoldade@peakpeak.com'; 'RANEY@VOLPE.DOT.GOV'
Subject: EPA APPROVED CONDITIONAL: LB-000015, LB-000016, and LB-000017

Richard,
Mark will modify LB-000015, 16 & 17 to indicate that the Effective Date is: Historical.
EPA approves these mods with this changed completed.

"Beckham, Richard" <BeckhamRE@cdm.com> To: Mary Goldade/EPR/R8/USEPA/US@EPA, '"RANEY@VOLPE.DOT.GOV"' <RANEY@VOLPE.DOT.GOV>, '"mgoldade@peakpeak.com"' <mgoldade@peakpeak.com>
cc: "Autio, Anni" <AutioAH@cdm.com>
Subject: FW: LB-000015, LB-000016, and LB-000017
04/01/03 08:47 AM

For your review and approval.

- Richard Beckham

-----Original Message-----
From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]
Sent: Monday, March 31, 2003 6:11 PM
To: Beckham, Richard

Subject: LB-000015, LB-000016, and LB-000017

Richard,

These should be final. The only recent revision is the addition of the Effective Date. These need to go to Mark and Mary for their final blessing.
<<LB-000015(rev 3_31_03).doc>> <<LB-000016 rev. (3_31_03).doc>>
<<LB-000017 rev(3_31_03).doc>>

R.K. Mahoney
Senior Analyst
Special Projects Coordinator
EMSL Analytical, Inc.
Westmont, NJ
800.220.3675, x1218
rmahoney@emsl.com

(See attached file: LB-000015(rev 3_31_03).doc)(See attached file: LB-000016 rev. (3_31_03).doc)(See attached file: LB-000017 rev(3_31_03).doc)

Request for Modification
To
Laboratory Activities
LB-000019

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable):TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: [All TEM Methodologies]

Requester: R. K. Mahoney Title: Senior Analyst/Special Projects Coordinator
Company: EMSL Analytical, Inc. Date: 21 January 2003

Description of Modification:
Clarification of bench sheet recording format for grid openings in which no countable structures are recorded.

Reason for Modification:
The electronically deliverable spread sheet for TEM analysis developed for the Libby project requires "ND" (None Detected) to be entered for grid openings in which no countable structures are recorded. The ND code has been used on all electronic deliverables for the Libby project. The code "NSD" (No Structure Detected) has been used on hand written bench sheets up until this date. As of 21 January 2003, "ND" will be used on the bench sheets as well as the electronically deliverables.

Potential Implications of this Modification:
There are no potential negative implications resulting from this clarification of terms.

Laboratory Applicability (circle one): All [Individual](s) EMSL Analytical, Inc.

Duration of Modification (circle one):
Temporary Date(s):
Analytical Batch ID:
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

[Permanent] (Complete Proposed Modification Section) Effective Date: 21 January 2003
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: R. K. Mahoney Date: 27 March 2003
(Laboratory Manager or designate)

Project Review and Approval: Mark Raney Date: 7 March 2003
(Volpe: Mark Raney)

Approved By: Mary Goldade Date: 7 March 2003
Title: EPA Regional Chemist
(USEPS: Mary Goldade)

Mahoney, Ron

From: Raney, Mark [RANEY@VOLPE.DOT.GOV]
Sent: Friday, March 07, 2003 2:50 PM
To: 'Beckham, Richard'; 'Charlie LaCerra'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio, Anni; Raney, Mark; 'brattin@syrres.com'; 'Goldade.mary@EPAmail.epa.gov'; Montera, Jeff
Subject: RE: MOD LB-000019

I find Laboratory Request for Modification # LB-000019 acceptable as written and here by provide Volpe approval to this MOD.

Richard, Please make sure MOD ID#s get inserted onto the mod forms themselves (not just the file ID), so you will be able to identify the IDs based upon hardcopy alone. Also, even though this MOD is applicable to an individual lab, all MODs are to be forwarded to all labs for informational purposes and to give them an opportunity to provide comments. All labs however are REQUIRED to provide comments to only MODs that are applicable to all labs.

Mark Raney
Environmental Engineer

US DOT / Volpe Center
Environmental Engineering Division, DTS-33
phone: 617-494-2377
cell: 617-694-8223
fax: 617-494-2789
raney@volpe.dot.gov

-----Original Message-----
From: Beckham, Richard [mailto:BeckhamRE@cdm.com]
Sent: Thursday, March 06, 2003 9:54 AM
To: 'Charlie LaCerra'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio, Anni; 'Raney@volpe.dot.gov'; 'brattin@syrres.com'; 'Goldade.mary@EPAmail.epa.gov'; Montera, Jeff
Subject: MOD LB-000019

This MOD impacts only EMSL. For your review and comment:

<<LB-000019.doc>>
- Richard Beckham

Mahoney, Ron

From: Mary Goldade [mgoldade@peakpeak.com]
Sent: Friday, March 07, 2003 12:29 PM
To: Raney, Mark
Cc: Jeff G. Montera; rmahoney@emsl.com; Autio, Anni; William Brattin; Goldade.Mary@epamail.epa.gov
Subject: Re: MOD LB-000019

I agree that this mod form is acceptable, and should be discussed on the next lab call to be certain similar issues are not encountered at other labs.
Mary
—— Original Message ——
From: "Raney, Mark" <RANEY@VOLPE.DOT.GOV>
To: "'Goldade, Mary (HOME)'" <mgoldade@peakpeak.com>
Sent: Friday, March 07, 2003 10:18 AM
Subject: FW: MOD LB-000019

>
> FYI
>
>
> -----Original Message-----
> From: Beckham, Richard [mailto:BeckhamRE@cdm.com]
> Sent: Thursday, March 06, 2003 9:54 AM
> To: 'Charlie LaCerra'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio, Anni; 'Raney@volpe.dot.gov'; 'brattin@syrres.com'; 'Goldade.mary@EPAmail.epa.gov'; Montera, Jeff
> Subject: MOD LB-000019
>
>
> This MOD impacts only EMSL. For your review and comment:
>
> <<LB-000019.doc>>
> - Richard Beckham
>
>
>

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Request for Modification
To
Laboratory Activities
LB-000028

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs

Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable):TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies

Requester: R. K. Mahoney Title: Senior Analyst / Special Projects Coordinator

Company: EMSL Analytical, Inc. Date: 17 June 2003

Description of Modification:

This is a clarification pertaining to the re-analysis of TEM samples when some of the originally read grid openings in a sample selected for re-analysis have become unreadable. In the event that more than half of the originally read grid openings have become unreadable, select the closest adjacent sample from the same sample delivery group with adequate intact grid openings for re-analysis. If half or less of the original openings on the sample selected are unreadable, make note in the Comments box in Data Entry 1 of the TEM EDD as to which grid openings are unreadable, and proceed with analysis of the original sample.

Reason for Modification:

This clarification is intended to provide more complete TEM re-analysis data.

Potential Implications of this Modification:

There are no negative implications to this clarification.

Laboratory Applicability (circle one): All Individual(s) _____

Duration of Modification (circle one):

Temporary Date(s): _____

Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: 17June 2003

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: R. K. Mahoney EMSL Date: 18 July 2003
(Laboratory Manager or designate)

Project Review and Approval: Mark Kanz Date: 7/18/03
(Volpe: Project Technical Lead or designate)

Approved By: [signature] Date: 6/24/03

Title: Project Chemist
(USEPA: Project Chemist or designate)

Mary Goldade

06/24/03 01:20 PM

To: "Beckham, Richard" <BeckhamRE@cdm.com>
cc: "Autio, Anni" <AutioAH@cdm.com>, 'Bill Egeland' <begeland@mastest.com>, "'Bob.Shumate@battaenv.com'" <Bob.Shumate@battaenv.com>, "'brattin@syrres.com'" <brattin@syrres.com>, 'Charlie LaCerra' <clacerra@emsl.com>, "'corbin77@atc-enviro.com'" <corbin77@atc-enviro.com>, "'dmazzaferro@mastest.com'" <dmazzaferro@mastest.com>, 'Gustavo Delgado' <gdelgado77@atc-enviro.com>, "'Garth B. Freeman'" <gfreeman@mastest.com>, "'jeanneorr@resienv.com'" <jeanneorr@resienv.com>, "'mgoldade@peakpeak.com'" <mgoldade@peakpeak.com>, "'m_szynskie@resienv.com'" <m_szynskie@resienv.com>, "'Naresh C. Batta'" <ncbatta@battaenv.com>, "'Raney@volpe.dot.gov'" <Raney@volpe.dot.gov>, "'rdemalo@emsl.com'" <rdemalo@emsl.com>, "'rhatfield@mastest.com'" <rhatfield@mastest.com>, "'rmahoney@emsl.com'" <rmahoney@emsl.com>, 'Shu-Chun Su' <scsu@delanet.com>, 'William Longo' <wlongo@mastest.com>
Subject: Re: EPA Approved w/ revisions MOD LB-000028

EPA approves Mod LB-000028 with revisions as attached.

LB-000028 (MG 6-24-03).

Mary Goldade
Regional Superfund Chemist
U.S. Environmental Protection Agency, Region 8
999 19th Street, Suite 300
Mail Code: 8EPR-PS
Denver, CO 80202

Phone: (303) 312-7024
Fax: (303) 312-6065
email: goldade.mary@epa.gov

"Beckham, Richard" <BeckhamRE@cdm.com>

"Beckham, Richard" <BeckhamRE@cdm.com>

06/23/03 08:42 AM

To: 'Charlie LaCerra' <clacerra@emsl.com>, 'Charlie LaCerra' <clacerra@emsl.com>, "'jeanneorr@resienv.com'" <jeanneorr@resienv.com>, "'rdemalo@emsl.com'" <rdemalo@emsl.com>, "'rmahoney@emsl.com'" <rmahoney@emsl.com>, 'William Longo' <wlongo@mastest.com>, "'rhatfield@mastest.com'" <rhatfield@mastest.com>, 'Bill Egeland' <begeland@mastest.com>, "'Bob.Shumate@battaenv.com'" <Bob.Shumate@battaenv.com>, "'Naresh C. Batta'" <ncbatta@battaenv.com>, 'Shu-Chun Su' <scsu@delanet.com>, "'corbin77@atc-enviro.com'" <corbin77@atc-enviro.com>, 'Gustavo Delgado' <gdelgado77@atc-enviro.com>, "'Garth B. Freeman'" <gfreeman@mastest.com>, "Autio, Anni" <AutioAH@cdm.com>, "'Raney@volpe.dot.gov'" <Raney@volpe.dot.gov>, "'brattin@syrres.com'" <brattin@syrres.com>, Mary Goldade/EPR/R8/USEPA/US@EPA, "'dmazzaferro@mastest.com'" <dmazzaferro@mastest.com>, "'mgoldade@peakpeak.com'" <mgoldade@peakpeak.com>, "'m_szynskie@resienv.com'" <m_szynskie@resienv.com>
cc:

Subject: MOD LB-000028

This MOD impacts all labs. For your review and comment.

- Richard Beckham

<<LB-000028.doc>>

From: "LaCerra, Charles" <CLaCerra@EMSL.com>
To: "Carr, Kim" <KCarr@EMSL.com>; "EMSL Mobile Lab - Asbestos" <mobileasbestoslab@EMSL.com>
Sent: Friday, July 18, 2003 5:57 AM
Attach: LB-000025_rev (MG 6-04-03 email).doc; LB-000027 (MG 6-24-03).doc; LB-000028 (MG 6-24-
Subject: FW: MODs: LB-000025, 26, 27 & 28

-----Original Message-----
From: Raney, Mark [mailto:RANEY@VOLPE.DOT.GOV]
Sent: Friday, July 18, 2003 7:53 AM
To: 'Beckham, Richard'; Autio, Anni
Cc: 'Goldade, Mary'; 'Goldade, Mary (HOME)'; 'Orr, Jeaane at Reservoir Env'; 'Mahoney, Ron'; 'Demalo, Rob (EMSL)'; 'LaCerra, Charles'
Subject: MODs: LB-000025, 26, 27 & 28

Richard,

LB-000025 (EMSL): Volpe provided approval (with revisions) on 6/18/03 & EPA approved on 5/14/03 (see emails and attachment below). I have yet to see a final version for signature. EMSL should finalize, sign and distribute for signature.

LB-000026 (EMSL): Approved and signed by both Volpe and EPA.

LB-000027 (RESI): MOD provided on 6/23/03 via Richard Beckham, Approved by EPA (with revisions) on 6/24/03. Volpe concurs with EPA and herby provides approval with EPA's revisions (see attached). RESI should finalize, sign and distribute for signature.

LB-000028 (EMSL): MOD provided on 6/23/03 via Richard Beckham, Approved by EPA (with revisions) on 6/24/03. Volpe concurs with EPA and herby provides approval with EPA's revisions (see attached). EMSL should finalize, sign and distribute for signature.

Please let me know if anyone has any questions.

Mark.

-----Original Message-----
From: Beckham, Richard [mailto:BeckhamRE@cdm.com]
Sent: Wednesday, July 16, 2003 5:30 PM
To: 'RANEY@VOLPE.DOT.GOV'; Autio, Anni
Subject: MOD Status

For MODs 27 and 28, I have email approvals from EPA, but have not been able
to locate approvals from Volpe. CDM received a hardcopy of 27 with an original signature from RESI, that was subsequently forwarded to Volpe on
7/8/3. (Did I miss an approval email?) To my knowledge, a hardcopy of 28
has not been prepared.

- Richard Beckham

-----Original Message-----
From: Raney, Mark
Sent: Wednesday, June 18, 2003 10:56 AM
To: 'Mahoney, Ron'
Cc: 'Anni Autio'; 'Mary Goldade'
Subject: RE: EPA Markups: MOD LB-000025

Ron,

I concur with Mary's comments below. I provide Volpe's approval for MOD LB-000025 with Mary's changes and the addition of an estimate of the number of samples involved (i.e,. < 20).

Thanks,

Mark.

-----Original Message-----
From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]
Sent: Wednesday, June 04, 2003 9:27 AM
To: 'Mark Raney'

Cc: 'Anni Autio'; 'Mary Goldade'; CDM STAFF
Subject: FW: EPA Markups: MOD LB-000025

Mark,

Do you have any other comments for this mod? Mary asked for an estimate of
the number of samples involved, and we agreed on < 20. The number is more
likely < 10, but we've deceided to err on the conservative side.

If I can get your input, we can put this one to bed.

R.K. Mahoney
Senior Analyst
Special Projects Coordinator
EMSL Analytical, Inc.
Westmont, NJ
800.220.3675, x1218
rmahoney@emsl.com

-----Original Message-----
From: Mary Goldade [mailto:mgoldade@peakpeak.com]
Sent: Wednesday, May 14, 2003 6:32 PM
To: Beckham, Richard; 'Charlie LaCerra'; jeanneorr@resienv.com; rdemalo@emsl.com; rmahoney@emsl.com; 'William Longo'; rhatfield@mastest.com; 'Bill Egeland'; Bob.Shumate@battaenv.com; 'Naresh C. Batta'; 'Shu-Chun Su'; corbin77@atc-enviro.com; 'Gustavo Delgado'; 'Garth B. Freeman'; Autio, Anni; Raney@volpe.dot.gov; brattin@syrres.com; Goldade.mary@EPAmail.epa.gov; dmazzaferro@mastest.com; m_szynskie@resienv.com
Subject: EPA Markups: MOD LB-000025

Suggested changes to the MOD are attached.
Ron-Do you already have in hand an estimate regarding the actual number of
samples this affects (i.e., are you able to quantify the term "few/limited"?)
Thanks,
Mary

----- Original Message -----
From: "Beckham, Richard" <BeckhamRE@cdm.com>
To: "'Charlie LaCerra'" <clacerra@emsl.com>; <jeanneorr@resienv.com>; <rdemalo@emsl.com>; <rmahoney@emsl.com>; "'William Longo'" <wlongo@mastest.com>; <rhatfield@mastest.com>; "'Bill Egeland'" <begeland@mastest.com>; <Bob.Shumate@battaenv.com>; "'Naresh C. Batta'" <ncbatta@battaenv.com>; "'Shu-Chun Su'" <scsu@delanet.com>;

<corbin77@atc-enviro.com>; "'Gustavo Delgado'"
<gdelgado77@atc-enviro.com>;
"'Garth B. Freeman'" <gfreeman@mastest.com>; "Autio, Anni"
<AutioAH@cdm.com>; <Raney@volpe.dot.gov>; <brattin@syrres.com>;
<Goldade.mary@EPAmail.epa.gov>; <dmazzaferro@mastest.com>;
<mgoldade@peakpeak.com>; <m_szynskie@resienv.com>
Sent: Wednesday, May 14, 2003 3:28 PM
Subject: MOD LB-000025

> This MOD impacts only EMSL. For your review and comment:
>
> <<LB-000025.doc>>
> - Richard Beckham

<<LB-000025_rev (MG 6-04-03 email).doc>> <<LB-000027 (MG 6-24-03).doc>> <<LB-000028 (MG 6-24-03).doc>>
>
>
>

Mary Goldade

07/29/03 01:57 PM

To: Anni Autio
cc: Mark Raney
cc:
Subject: LB-000027 & LB-000028 are signed and mailed

Anni & Joe,
I have mail you the original copiew of the mods LB-000027 & LB-000028.
Several of the email approval pages were not provided. I attached them.

Mary Goldade
Regional Superfund Chemist
U.S. Environmental Protection Agency, Region 8
999 19th Street, Suite 300
Mail Code: 8EPR-PS
Denver, CO 80202

Phone: (303) 312-7024
Fax: (303) 312-6065
email: goldade.mary@epa.gov

Request for Modification
to
Laboratory Activities
LB-000029b

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): [TEM-AHERA] [TEM-ISO 10312] PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 [ASTM D5755] EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Lynn Woodbury Title: Technical consultant
Company: Syracuse Research Corporation Date: December 7, 2006

Description of Modification:
Permanent clarifications to laboratory-based Quality Control (QC) sample analysis. The purpose of the attached is to standardize the frequency of analysis and procedures for interpretation of the results for laboratory-based Quality Control (QC) samples for TEM analyses of air and dust. The general concepts presented in this modification may also be used for soil and water, but specific details regarding the frequency and interpretation of laboratory QC samples will need to be adjusted for these media.

Reason for Modification:
This modification is needed to standardize the frequency with which different types of QC samples are prepared in different laboratories in the program, and to ensure that all results are evaluated in accord with a standard set of criteria.

Potential Implications of this Modification:
There are no potential negative implications resulting from this standardization of QC procedures.

Laboratory Applicability (circle one): [All] Individual(s) _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

[Permanent] (Complete Proposed Modification Section) Effective Date: _____
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

[Not Applicable] **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): _____

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 4/25/07
(Volpe Project Technical Lead or designate)

Approved By: _____ Date: 4/25/07
(USEPA Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

QC Sample Type Definitions

There are three categories of TEM laboratory QC samples: Blanks, Recounts, and Repreparations.

Blanks

Lab Blank (LB) – This is a TEM grid that is prepared from a new, unused filter by the laboratory and is analyzed using the same procedure as used for field samples.

Recounts

Recount Same (RS) – This is a TEM grid that is re-examined within the same laboratory and by the same microscopist who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Same TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Recount Different (RD) – This is a TEM grid that is re-examined within the same laboratory but by a different microscopist than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Different TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Interlab (IL) - This is a TEM grid that is re-examined by a microscopist from a different laboratory than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Interlab TEM analyses for air and dust will be selected in accord with the procedure presented in Attachment 2.

Verified Analysis (VA) – This is a recount of a TEM grid (same grid openings) performed in accord with the protocol for verified analysis as provided in NIST (1994) (provided as Attachment 3). Verified TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Repreparations

Repreparation (RP) – This is a TEM grid that is prepared from a new portion of the same filter that was used to prepare the original grid. Typically this is done within the same laboratory as did the original analysis, but a different laboratory may also prepare grids from a new piece of filter. Repreparations will be selected in accord with the procedure presented in Attachment 1.

Frequency

The minimum frequency for laboratory-based QC samples for TEM analyses (all media combined) shall be as follows:

QC Sample Type	Min. Frequency
Lab blank	4%
Recount same	1%
Recount different	2.5%
Verified analysis	1%
Repreparation	1%
Interlab	0.5%
Total	**10%**

Each laboratory should prepare and analyze lab blank, recount (same, different and verified), and repreparation samples at the minimum frequency specified in the table above. The selection procedure and laboratory SOP for the selection of samples for the purposes of recounts and repreparation are provided in Attachment 1. Samples for interlab comparisons will be selected by EPA's technical consultant (SRC) in accord with the selection procedure and laboratory SOP provided in Attachment 2.

Procedure for Evaluating QC Samples and Responses to Exceptions

The procedure for evaluating QC sample results varies depending on sample type. These procedures are presented below.

Note: The procedures for evaluating QC samples presented below are based in part on professional judgement and experience at the site to date. These procedures and rules for interpretation may be revised as more data are collected.

Lab Blanks.

There shall be no asbestos structure of any type detected in an analysis of 10 grid openings on any lab blank. If one or more asbestos structures are detected, the laboratory shall immediately investigate the source of the contamination and take immediate steps to eliminate the source of contamination before analysis of any investigative samples may begin.

Recounts.

All recount samples (same, different, verified, and interlab) will be evaluated by comparing the raw data sheets prepared by each analyst. Note that the raw data for samples must include sketches for both the initial and QC reanalysis, as described in modification LB-000030. All structure enumeration and measurements will adhere to the established project-specific documentation presented in LB-000016A and LB-000031A. The following criteria will be used to identify cases where results for LA structures are concordant (in agreement) or discordant (not in agreement). These LA criteria were established by microscopists experienced in the analysis of Libby amphibole asbestos, and serve as an initial attempt at review criteria developed using their professional experience. As the database continues to grow and we learn more, these criteria may be revisited and revised. Changes to the criteria for LA structures will be accompanied by scientific justification to support the change. Criteria for concordance on non-LA fibers (OA and C) fibers are the same as described in NIST (1994) (provided as Attachment 3).

Measurement parameter	Concordance Rule
Number of LA asbestos structures within each grid opening	For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10%.
Asbestos class of structure (LA, OA, C)	Must agree 100% on chrysotile vs. amphibole. For assignment of amphiboles to LA or OA bins, must agree on at least 90% of all amphibole structures.
LA Structure length	For fibers and bundles, must agree within 0.5 um or 10% (whichever is less stringent) For clusters and matrices, must agree within 1 um or 20% (whichever is less stringent)
LA Structure width	For fibers and bundles, must agree within 0.5 um or 20% (whichever is less stringent). For clusters and matrices, there is no quantitative rule for concordance.

Whenever a recount occurs in which there is one or more discordance, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will use the results of the validated analysis to determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc). Whichever analytical result is determined to be correct will be identified with the word "Confirmed" in the sample comment field of the electronic data reporting sheet. In the special case where the original and the reanalysis are both determined to have one or more areas of discordance, a third electronic data report will be prepared that contains the correct results. This will be identified as QA Type = "Reconciliation". The laboratory should maintain records of all cases of discordant results and of actions taken to address any problems, in accord with the usual procedures and requirements of NVLAP. In addition, each laboratory should notify the CDM Laboratory Manager of any significant exceptions and corrective actions through a job-specific (temporary) modification form. The CDM Laboratory Manager will ensure that appropriate Volpe and EPA representatives are notified accordingly.

Repreparations.
Repreparation samples will be evaluated by comparing the total counts for the original and the re-preparation samples. In order to be ranked as concordant, the results must not be statistically different from each other at the 90% confidence interval, tested using the statistical procedure documented in Attachment 4. Whenever an exception is identified, a senior analyst shall determine the basis of the discordant results, and if it is judged to be related to laboratory procedures (as opposed to unavoidable variability in the sample), the laboratory shall then take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, quantification of size, identification of types, etc).

Program-Wide Goals

While each laboratory shall monitor the results of the QC samples analyzed within their laboratory and shall take actions as described above, the overall performance of the program shall be monitored by assembling summary statistics on QC samples, combining data within and across laboratories. The program-wide goals shall be interpreted as follows:

QC Sample Type	Metric	Program-Wide Criteria		
		Good	Acceptable	Poor
Lab Blanks	% with ≥1 asbestos structures	0% - 0.1%	0.2% - 0.5%	>0.5%
Recounts	Concordance on LA count	>95%	85-95%	<85%
	Concordance on type (chrysotile vs. amphibole)	>99%	95%-99%	<95%
	Concordance on LA length	>90%	80%-90%	<80%
	Concordance on LA width	>90%	80%-90%	<80%
Repreps	Concordance on LA concentration/loading	>95%	90-95%	<90%

As the database continues to grow and we learn more, these project-wide goals may be revisited and revised. Changes to the project-wide goals will be accompanied by appropriate justification to support the change.

REFERENCES

NIST. 1994. Airborne Asbestos Method: Standard Test method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0. National Institute of Standards and Technology, Washington DC. NISTIR 5351. March 1994.

ATTACHMENT 1

Selection Procedure and Laboratory SOP for Recounts (RS, RD, VA) and Repreparations (RP)

Selection Procedure

As specified in the Frequency section above, the frequency of Recount Same (RS) should be 1%, the frequency of Recount Different (RD) should be 2.5%, the frequency of Verified Analyses (VA) should be 1%, and the frequency of Repreparations (RP) should be 1%, corresponding to a total within-laboratory QC frequency of 5.5% for these analysis types. This is approximately 1 QC sample per 20 field samples. Based on this frequency, it is possible to determine which laboratory job(s) will have one or more samples selected for recount analysis or repreparation.

For those laboratory jobs in which a recount or repreparation sample is to be selected, the analyst should record the total number of structures observed in each sample. The sample(s) selected for recount or repreparation should be those within the laboratory job with the highest number of structures per grid opening (GO) area examined (calculated as the number of GOs evaluated * the GO area). When selecting samples for repreparation, if possible, preferentially select samples in which the total number of GOs is 40 or less. Because repreparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, repreparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the selection of samples with 40 GOs or less will reduce analytical costs associated with repreparations. When selecting samples for recount, it is not necessary to impose a minimum or maximum number of GOs because concordance is evaluated on a GO and structure basis, rather than a concentration basis. If all samples within the laboratory job are non-detect, a non-detect sample may be selected. A non-detect sample should be preferentially selected, every 10^{th} selection.

This selection procedure will ensure that the recount analyses and repreparations yield a dataset best suited to assess concordance[1].

Laboratory SOP for Recount Analyses

1. For recount samples, re-analyze the selected sample in accord with the appropriate procedures for each type of recount (RS, RD, or VA). If more than 10 GOs were evaluated in the original analysis, the original analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the recount analysis. The original analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures per GO area examined, which may be analyzed in the event that a selected GO is damaged and cannot be re-evaluated.

2. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Recount Same", "Recount Different", or "Verified Analysis", as appropriate. Be sure that the grid and GO names match exactly with the names evaluated in the original analysis (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the original analyst or laboratory director to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).

3. If there is one or more discordant GOs between the original analysis and the recount analysis, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

[1] It should be noted that this selection procedure will tend to result in the preferential selection of samples with the highest air concentration/dust loading values. Thus, summary statistics based on laboratory QC samples may tend to be biased high.

4. Submit the recount TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

Laboratory SOP for Repreparations

1. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.

2. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the repreparation. Place the remaining grid in storage.

3. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".

4. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

ATTACHMENT 2

Selection Procedure and Laboratory SOP for Interlabs (IL)

Selection Procedure

1. On the 1st of each month, EPA's technical consultant (SRC) will compile a list of all samples for which air and dust TEM results (ISO+AHERA+ASTM) were uploaded into Libby V2 Database in the preceding month (e.g., on November 1st, specify a date range of Oct 1-31, 2005). The Libby V2 Database query will be based on the upload date rather than the analysis date to ensure that analyses with an upload in a different month as the analysis date were not excluded[2].

2. Identify the target number of air and dust interlab samples needed to meet the QC requirements for interlabs specified in the Frequency section above (0.5%). This is accomplished by multiplying the desired interlab frequency (0.5%) by the total number of air and dust analyses performed in the preceding month. For example, 178 TEM air analyses in October 2005 * 0.5% = 0.89 (which is rounded up to 1). At a minimum, at least one air and one dust sample will be selected for interlab analysis.

3. For each medium (air and dust), rank order the TEM analyses from the preceding month on the total number of LA structures per GO area examined (calculated as the number of GOs evaluated * the GO area). Selecting from analyses with a high number of LA structures per GO area examined increases the likelihood that the GOs evaluated as part of the interlab analysis will have one or more LA structures.

4. Exclude samples in which the total number of GOs is more than 40 GOs[3]. Exclude any samples that have already been selected for interlab evaluation previously.

5. Select the appropriate number of air and dust interlab samples from the available TEM analyses for which the total number of LA structures per GO area examined is higher than 0 (i.e., LA detects). If the total number of samples with LA detects is equal to the desired number of interlab samples, select all detected samples for interlab analysis. If the total number of samples with LA detects is less than to the desired number of interlab samples, select non-detect samples for interlab analysis. If the total number of samples with LA detects is higher to the desired number of samples, interlab samples will be selected to represent multiple laboratories, selecting those samples with the highest number of LA structures per GO examined first. EPA's technical consultant (SRC) will keep a running total of the number of samples selected by laboratory to ensure that the long-term frequency of interlabs for each laboratory is generally similar.

6. Submit list of selected interlab samples to the CDM Laboratory Manager.

7. Each month, the CDM Laboratory Manager will provide each laboratory with the list of samples selected for Interlab analysis.

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[2] Consider the case where the TEM analysis for sample X-12345 was performed on September 22 and the results were uploaded on October 3. The interlab selection query performed on October 1, if limited to all results analyzed from September 1-30, would not capture the results for X-12345 because they had not yet been uploaded. The interlab selection query performed on November 1, limited to all results analyzed from October 1-31, would also not capture the results for sample X-12345 because the analysis date is outside of the specified range.

[3] Because all interlabs will be reprepared, these interlab repreparation samples will also be evaluated for concordance with the original sample. Because repreparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, repreparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the focusing on samples with 40 GOs or less will reduce analytical costs associated with repreparations.

At the Originating Laboratory:

1. Upon receipt of the interlab sample list from the CDM Laboratory Manager, locate the appropriate sample filter. If less than ¼ of the sample filter is available, contact the CDM Laboratory Manager to identify an interlab replacement sample.

2. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.

3. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the repreparation. Place the remaining grid in storage.

4. Record the orientation of each grid using the instructions for grid orientation specified in NVLAP (see Attachment 5).

5. When performing the TEM analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.

6. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".

7. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

8. Identify which laboratory will perform the interlab analysis in accord with the following table:

Originating Lab	Lab for Interlab Sample #1	Lab for Interlab Sample #2	Lab for Interlab Sample #3	Lab for Interlab Sample #4	Lab for Interlab Sample #5	Lab for Interlab Sample #6...
Hygeia	Batta	MAS	RESI	EMSL-L	EMSL-W	Repeat... (beginning with the Lab identified for Sample #1)
Batta	MAS	RESI	EMSL-L	EMSL-W	Hygeia	
MAS	RESI	EMSL-L	EMSL-W	Hygeia	Batta	
RESI	EMSL-L	EMSL-W	Hygeia	Batta	MAS	
EMSL-L	EMSL-W	Hygeia	Batta	MAS	RESI	
EMSL-W	Hygeia	Batta	MAS	RESI	EMSL-L	

EMSL-L = EMSL, Mobile Lab in Libby
EMSL-W = EMSL, Westmont

9. If more than 10 GOs were evaluated in the repreparation analysis, the repreparation analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the interlab analysis. The repreparation analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures, which may be analyzed in the event that the selected GO is damaged and cannot be re-evaluated.

10. Ship the grid(s) for the interlab sample to the appropriate laboratory using standard chain of custody procedures. For each interlab sample, include a list of which GOs should be evaluated for each grid. The names of the grid and GOs provided on the chain of custody form should match exactly with those recorded in the original TEM data recording spreadsheet (including dashes, underscores, and spaces).

11. After the interlab laboratory has completed the interlab analysis, it will request copies of the hard copy laboratory benchsheet(s), the grid opening sketches, and TEM file for each interlab sample.

12. If areas of discordance are noted, the senior laboratory analyst from the interlab laboratory will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

At the Interlab Laboratory:

1. For each grid provided for interlab analysis, place the grid into the TEM grid holder ensuring that the grid orientation matches that which was specified by the originating laboratory (see Attachment 5 for details).
2. For the 10 GOs identified for interlab analysis, perform TEM analysis using the analysis method and counting rules specified on the chain of custody. Be sure that the grid and GO names match exactly with the names provided on the chain of custody (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the originating laboratory to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).

3. When performing the TEM interlab analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.

4. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Interlab".

5. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

6. Contact the originating laboratory to request copies of the hard copy laboratory benchsheet(s), grid opening sketches, and TEM file for each interlab sample.

7. Perform a verified analysis using the procedures presented in NIST (1994) (provided as Attachment 3).

8. Assess the between-laboratory concordance, both on a GO-by-GO basis and on a structure-by-structure basis, using the Libby-specific recount concordance rules. If areas of discordance are noted, the senior laboratory analyst will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

9. Summarize the results of the verified analysis and document any changes in laboratory procedures or analyst training that were implemented to address noted discordances. Provide a copy of this report to EPA Chemist and the CDM Laboratory Manager.

10. Ship the grid(s) back to the originating lab.

ATTACHMENT 3

Airborne Asbestos Method:
Standard Test Method for Verified Analysis of Asbestos
by Transmission Electron Microscopy-Version 2.0.
NIST (1994)

NISTIR 5351

Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0

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NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
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Preface

This Interagency Report (IR) is one of a series of IRs that will form the basis of a method for analysis of airborne asbestos by transmission electron microscopy. The form and style of the American Society for Testing and Materials (ASTM) was adopted as a standard format for this series of reports.

1. Scope

1.1 This test method describes a procedure for verified analysis of asbestos by transmission electron microscopy.

1.2 The method is applicable only when sufficient information has been collected during the analyses of a grid square so that individual asbestos structures can be uniquely identified.

1.3 The method is written for the analysis of a grid square by two TEM operators but can be used for more than two operators with slight modifications. Due to the analysis of a grid square by more than one TEM operator, the test method can be applied only when contamination and beam damage of particles are minimized. The two TEM operators can use the same TEM for the analysis or the analyses can be done on different TEMs (in the same or in different laboratories).

1.4 The method can be used with any set of counting rules applied by all analysts. Though the method describes verification of asbestos particles, the method can also be used for verification of analyses of nonasbestos particles if all analysts use the same counting rules.

2. Terminology

2.1 *Definitions:*

2.1.1 *TEM*--transmission electron microscope.

2.1.2 *grid square, grid opening*--an area on a grid used for analysis of asbestos by transmission electron microscopy.

2.1.3 *verified analysis*--a procedure in which a grid opening is independently analyzed for asbestos by two or more TEM operators and in which a comparison and evaluation of the correctness of the analyses are made by a verifying analyst. Detailed information -- including absolute or relative location, a sketch, orientation, size (length, width), morphology, analytical information and identification -- is recorded for each observed structure.

2.1.3.1 *Discussion*--Verified analysis can be used to determine the accuracy of operators and to determine the nature of problems that the analyst may have in performing accurate analyses. Verified counts can be used to train new analysts and to monitor the consistency of analysts over time.

2.2 *Description of Terms Specific to This Standard:*

2.2.1 *counting rules*--rules used to determine the amount of asbestos present in an asbestos- containing sample. Counting rules are a part of most methods for analysis of asbestos by transmission electron microscopy including the AHERA method and the ISO method (see definitions below).

2.2.2 *AHERA method*[1]--procedure for analysis of asbestos by transmission electron microscopy developed by the Environmental Protection Agency with subsequent modifications by the National Institute of Standards and Technology.

2.2.3 *ISO method*[2]--procedure for analysis of asbestos by transmission electron microscopy developed by the International Standards Organization.

2.2.4 *particle*--an isolated collection of material deposited on a grid or filter.

2.2.5 *structure*--a particle or portion of a particle that contains asbestos and that is considered countable under the method used for asbestos analysis. A structure is a basic unit used in many methods of asbestos analysis to report the amount of asbestos present in a particle.

2.2.6 *TEM operator, TEM analyst*--person that analyzes a grid square by transmission electron microscopy to determine the presence of asbestos.

2.2.7 *verifying analyst*--person that compares the analyses of a grid square by two or more TEM operators. The reported asbestos is compared on a structure-by-structure basis by the verifying analyst. Structures that are not matched are relocated and reanalyzed by the verifying analyst. The verifying analyst is

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[1]Code Fed. Reg. **1987**, *52* (No. 210), 41826-41905.

[2]ISO 10312 **1993**, in press.

preferably not one of the TEM operators. If this cannot be avoided, the job of verifying analyst should be rotated between the TEM operators.

2.2.8 *TEM analysis form*--form on which the analysis of a grid square is recorded. The information recorded for a verified analysis should include at least a sketch of the structure and information related to the absolute or relative location, size, identification and analytical data for the reported structures.

2.2.9 *report form*--form on which the evaluation of verified analyses is summarized. The form should be identical to or include all information given in Figure X1.1 of Appendix X1.

2.2.10 *SR (structures reported)*--the number of structures reported by a TEM analyst.

2.2.11 *TP (true positive)*--structure that is: 1) reported by both TEM operators or 2) reported by one operator and confirmed by the verifying analyst, or 3) reported by neither TEM operator but is found by the verifying analyst. The three types of true positives are discussed in the next three terms.

2.2.12 *TPM (true positive-matched)*--structure that is reported on the TEM analysis forms of both TEM operators.

2.2.12.1 *Discussion*--To qualify as a match, the structures should be comparable in the following characteristics: 1) absolute or relative location, 2) appearance in the sketch, 3) orientation, 4) size (length, width), 5) morphology (shape, hollow tube), 6) analytical information (chemistry and/or diffraction data), and 7) identification. In addition, the structures should be reported as countable by both analysts.

2.2.13 *TPU (true postive-unmatched)*--structure that is reported on the TEM analysis form of only one operator and that is confirmed as countable by the verifying analyst.

2.2.14 *TPV (true positive found by verifying analyst)*--structure not found by the two TEM operators but found by the verifying analyst.

2.2.15 *TNS (total number of structures)*--the number of structures determined to be in a grid opening by verified analysis of the grid opening. This value corresponds to the number of unique true positives found by the TEM operators and the verifying analyst.

2.2.15.1 *Discussion*--The value for the total number of structures is not necessarily the actual number on the grid square because both the TEM analysts and the verifying analyst may have missed one or more structures. The probability of a missed structure, however, decreases with an increased number of analysts.

2.2.16 *FN (false negative)*--structure that has not been reported as countable by one of the TEM analysts. False negatives can be divided into two categories-type A and type B as discussed in the next two terms.

2.2.17 *FNA (false negative-type A)*--false negative that was recorded on a TEM analyst's TEM analysis form but not reported as a structure. Some reasons for this type of false negative include: 1) structure misidentified as nonasbestos, 2) confusion with the counting rules, 3) incorrect length determination.

2.2.18 *FNB (false negative-type B)*--false negative that was not recorded on a TEM analyst's TEM analysis form. A reason for this type of false negative is that a structure was missed by an analyst.

2.2.19 *FP (false positive)*--reported particle that is incorrectly identified as a structure. Some reasons for false positives include: 1) structures counted more than one time, 2) materials misidentified as asbestos, 3) confusion with the counting rules, 4) incorrect length determination.

2.2.20 *TN (true negative)*--reported particle that is correctly characterized as zero structures.

2.2.21 *NL (not located structure)*--structure reported on one TEM analyst's TEM analysis form that cannot be located by the verifying analyst.

2.2.21.1 *Discussion*--The value for NL should be zero for most verified analyses, especially if the grid has not been removed from the TEM between the two analysts' counts. If, however, a grid has been removed from an instrument, there is a small possibility of fiber loss.

2.2.22 *AMB (ambiguous structure)*--a structure that 1) is identified as a structure by only one TEM operator and 2) is found by the verifying analyst but cannot be unambiguously identified as a structure due to beam damage, contamination, or other factors.

3. Significance and Use

3.1 The analysis of asbestos by transmission electron microscopy is important for the determination of the cleanliness of air or water and for research purposes. Verified analyses provide more accurate values for the concentration of asbestos on a grid opening than obtained by other methods. The accuracy should increase with an increased number of analysts participating in the verified count.

3.2 The test method can be used as part of a quality assurance program for asbestos analyses and as a training procedure for new analysts. The values for TP/TNS and FP/TNS can be plotted *vs* time on control charts to show improvements or degradations in the quality of the analyses. Experienced analysts should attain TP/TNS values ≥ 0.85 and FP/TNS values ≤ 0.05. The test method can be used to characterize the types and, in many cases, the causes of problems experienced by TEM analysts.

3.3 The average of values obtained for TP/TNS and FP/TNS can be used to determine the analytical uncertainty for routine asbestos analyses.

4. Procedure

NOTE 1-- This test method involves two TEM operators and a verifying analyst. The steps discussed in items 4.1 and 4.2 are to be followed by the person coordinating the analyses by the TEM operators. This person can be one of the TEM operators, the verifying analyst or an independent person (e.g., a quality assurance officer). The steps discussed starting with item 4.3 are to be followed by the verifying analyst.

4.1 Obtain analyses of a grid square for asbestos by two TEM operators. Conduct the analyses independently so that the second operator has no knowledge of the results obtained by the first operator.

4.1.1 Require that the TEM operators record on the TEM analysis form information related to the absolute location of the structures or conduct analyses so that the relative location of the structures can be compared.

NOTE 2– The absolute location of the structures can be recorded by various means including use of a digital voltmeter or computer readable stepping motors to record the position of a structure. To preserve information about the relative location of the reported structures, the analyses must be conducted so that both analysts: 1) orient the grid in the TEM in the same fashion, 2) start the analysis from the same corner of the grid square, 3) initially scan in the same direction, and 4) scan the grid square in parallel traverses.

4.1.2 Require that the TEM operators record on the TEM analysis form a sketch of the structure, the dimensions of the structure, analytical data and whether the structure is countable. The sketch of the structure should include any nearby features that could aid in subsequent identification - for instance, nearby particles, sample preparation features or grid bars.

4.2 Submit the analyses of the two TEM operators to the verifying analyst.

NOTE 3– The remainder of this section describes procedures to be followed by the verifying analyst. The procedure for comparison of the TEM analysis forms is given in items 4.3-4.6 and examples of comparisons of count sheets are given in Figs. X2.1-X2.9 of Appendix 2. Appendix 3 contains a summary of the comparison process (Fig. X3.1) and a flow chart for comparison of structures in the TEM (Fig. X3.2). The procedure for completion of the report form is given in item 4.7.

4.3 Compare the two TEM analysis forms on a structure-by-structure basis. If a match of asbestos structures is observed, label both sketches with a TPM(number) either in the sketch box or in a column specifically designated for verified counts. An example is given in Fig. X2.1 of Appendix X2.

NOTE 4-- The next step in the procedure (item 4.4) is optional. The most prudent approach is to examine unmatched structures in the TEM (item 4.5).

4.4 Determine if the status of any of the unmatched structures can be unambiguously decided by examining the TEM analysis forms. If there is ambiguity in determining the status of a structure, the verifying analyst must examine the structure in the TEM as described in items 4.5-4.6. The comparison of TEM analysis forms and labelling of unmatched structures can be relatively straight foward as shown in Fig. X2.2 - X2.4 of Appendix X2 or more complex as described in the next item.

4.4.1 For most cases, the identification of true positives, false positives and false negatives can be done on a structure-by-structure basis. This cannot be done, however, in cases where analysts determine different numbers of countable structures in an asbestos-containing particle. In such cases, both analysts should be assigned one TPM(number) for identifying the particle as containing countable asbestos. The remaining structures are assigned TPU, FP or FN depending on the particular situation. Examples of such cases are given in Fig. X2.5 and Fig. X2.6 of Appendix X2.

4.5 Determine the status of any remaining unlabelled structures by examining the grid square in the TEM. Examples of TEM analysis forms containing structures that must be examined by transmission electron microscopy are given in Figs. X2.7 - X2.9 of Appendix 2. For each unlabelled structure requiring examination by transmission electron microscopy, follow items 4.5.1-4.5.7 and 4.6 until the structure is labelled. If there is another unlabelled structure, go back to item 4.5.1 and repeat the procedure. Continue until all structures are labelled. A summary flow chart for examination by TEM is given in Fig. X3.2. The procedure and flowchart do not cover the counting discrepancy discussed in item 4.4.1. If such a situation is recognized, the verifying analyst should follow the procedure given in item 4.4.1 and in the examples in Figs. X2.5 and X2.6.

NOTE 5-- The procedure in items 4.5.1-4.5.7 should cover the great majority of cases encountered when attempting to determine the status of the structures. There may, however, be more complex situations not covered in the procedure. If so, the verifying analyst should apply the basic principles outlined in items 4.5.1-4.5.7 and 4.4.1.

4.5.1 Determine if the reported structure can be located. If the structure cannnot be found, label the reported structure NL (place the label next to the sketch or in a column specifically designated for verified analyses).

4.5.2 If the reported structure is found, determine if a judgement can be made as to its countability. If the structure cannot be judged as to its countability due to beam damage, contamination or other factors, label the reported structure AMB.

4.5.3 If a judgement can be made as to the countability of the reported structure, determine if the structure is countable. If the reported structure is not countable, label it FP(number). A unique number is given to the FP label so that it can be specifically referred to in the report form. Optional: Check the other analyst's TEM analysis form. If the other analyst sketched the particle and correctly reported it as noncountable, label the particle TN(number). Note: The values for TN are not recorded on the report form.

4.5.4 If the reported structure is correctly identified as a structure, determine if it was reported as countable elsewhere on the same analyst's TEM analysis form (i.e., the analyst counted the structure twice). If it is a duplicate, label the reported structure FP(number).

4.5.5 If the reported structure is not a duplicate, label the structure TPU(number).

4.5.6 Determine if the other TEM operator recorded a sketch of the structure. If the other TEM operator did not report the structure on his/her TEM analysis form, place an FNB(number) on their TEM analysis form in the approximate location where the structure should have been found. The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.5.7 If the other TEM operator recorded a sketch of the structure, label the sketch with an FNA(number). The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.6 Countable asbestos structures reported by neither TEM operator but found by the verifying analyst in the course of examining a grid square should be recorded on a separate TEM analysis form and labelled

TPV(number). The TEM operators should be assigned an FNA(number) or FNB(number) as described in items 4.5.6-4.5.7.

4.7 Complete the report form as described in items 4.7.1-4.7.10.

4.7.1 Complete the heading of the report form and fill in the initials or names of the two TEM operators on the first line of the report form table.

4.7.2 Count the number of asbestos structures obtained by each analyst and enter the value as SR (structures reported) on the report form.

4.7.3 Determine the number of true positives that are matched (TPM), the number of true positives that are unmatched (TPU) and the total number of true positives (TP) obtained for each TEM operator on the grid square and enter the values on the report form.

4.7.4 Determine and record on the report form the number of true positives found by the verifying analyst (TPV).

4.7.5 Determine and record on the report form the total number of structures (TNS) on the grid square.

4.7.6 Determine and record on the report form for each operator the following: 1) the number of false positives (FP), 2) the number of false negatives (FN), 3) the number of false negatives of type A and type B (FNA, FNB), 4) the number of structures that were not located (NL) and 5) the number of ambiguous structures (AMB).

4.7.7 Determine and record the values for TP/TNS, FP/TNS to two decimal places.

4.7.8 List on the report form the suspected reasons for the false positives obtained by each analyst. Some examples would be as follows: incorrect length measurement, structures counted twice, problem with interpretation of the counting rules, misidentification of a structure.

4.7.9 List on the report form the suspected reasons for false negatives (FNA and FNB). Some examples would be: incorrect length measurement, problem with interpretation of the counting rules, misidentification of material as asbestos, possible loss of sense of direction, and insufficient overlap of traverses.

4.7.10 Append any other relevant comments to the report form (quality of the preparation, etc.).

4.8 Check the numbers on the report form using the equations given in the calculation section.

5. Calculation

5.1 The values on the report form should be consistent with the following equations:

For both analyses:

$$TNS = TPM + TPU(\text{Operator 1}) + TPU(\text{Operator 2}) + TPV$$

For a given analysis:

$$SR = TP + FP + NL + AMB$$

$$TP = TPM + TPU$$

$$FN = FNA + FNB$$

$$TNS = TP + FN$$

$$1 = TP/TNS + FN/TNS$$

6. Precision and Bias

6.1 To determine the precision of the method, independent verified analyses were conducted by operators in two laboratories on a set of 21 grid squares. The mean value for TNS for the data set was 16.2 structures/grid square and the pooled standard deviation of the pairs of verified count determinations was 1.12 structures/grid square. The confidence at approximately the 95% level (2 standard deviations) of a reported verified count value in this data set is 2.24 structures/grid square or 13.9% of the mean value for TNS. We use 13.9% as an estimate of the imprecision of the method.

NOTE 6-- The differences in the values obtained for the independent verified analyses described in item 6.1 are, for the most part, due to differences in interpretation of the counting rules. The structures analyzed in the study were complex and therefore the imprecision estimate discussed above likely represents an upper bound to the imprecision for the method.

6.2 The bias in the method will vary depending upon interpretation of the counting rules used in the analysis by the TEM operators and verifying analyst.

7. Keywords

7.1 asbestos; quality assurance; transmission electron microscopy; verified analysis

APPENDIXES

(Nonmandatory Information)

X1. TEST REPORT FORM

Fig. X1.1 The following format is suggested for use by the verifying analyst to report the comparison of the TEM operators' TEM analysis forms.

Grid box: _____ Date: _____

Grid slot: _____ Verifying Analyst: _____

Grid square: _____

	Analysis 1	Analysis 2
TEM Operator		
Structures Reported (SR)		
True Positives (TP)		
*TPM		
TPU		
*TPV		
*Total # Structures (TNS)		
False Positives (FP)		
False Negatives (FN)		
FNA		
FNB		
Not Located (NL)		
Ambiguous (AMB)		
TP/TNS		
FP/TNS		

*The values for these items will be the same for both analyses.

Test Report Form (continued)

1) List details of suspected reasons for false positives. For each analyst describe reasons for FP1, FP2, FP3, etc. Note - it may not be possible to determine the reason for false positives for some structures.

2) List details of suspected reasons for false negatives (type A and type B). For each analyst describe reasons for FNA1, FNA2, etc.; FNB1, FNB2, etc. Note - it may not be possible to determine the reasons for false negatives for some structures.

X2. EXAMPLES OF COMPARISONS OF TEM ANALYSIS FORMS

[Note: The TEM analysis forms shown in the examples are abbreviated and do not contain analysis information. The AHERA counting rules (1987) were used for all analyses.]

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr

Analyst 2

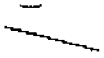
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.1 Example of matching structures on two TEM analysis forms (refer to item 4.3 of the procedure). Three structures on a grid square were found by both analysts. The relative order of the last two structures is different on the two TEM analysis forms; this may be due to the nature of the traverses by the analysts. Matching structures are indicated by TPM(number).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		FP1	1	Chr

Analyst 2

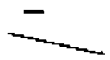
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.2 Example of determining the status of an unmatched structure from TEM analysis forms (refer to item 4.4 of the procedure). Three of the structures match in the two analyses. The last structure of analyst 1 is unmatched but can be seen from the TEM analysis form to be a duplicate of the second structure obtained by the same analyst (the two structures have the same identification, dimensions, orientation and a similar nearby particle). The duplicate structure is therefore assigned an FP1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

Analyst 2

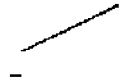
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		FNA1	0	Chr

Fig. X2.3 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the structure. However, analyst 2 has reported that the particle is not a structure (the cause of this oversight is not known). Analyst 1 is assigned a TPU1 and analyst 2 an FNA1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		FP1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Fig. X2.4 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the particle on both TEM analysis forms. However, analyst 1 has reported that the particle is a structure (the cause of this oversight is not known). Analyst 1 is assigned an FP1 and analyst 2 a TN1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1	0.6		TPM1 FNA1	1	Chr

Analyst 2

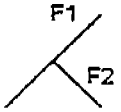
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
1	0.1	F1	TPM1	1	Chr
0.6	0.1	F2	TPU1	1	Chr

Fig. X2.5 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one countable structure and analyst 2 has reported two countable structures. Under the AHERA counting rules, analyst 2 is correct. The structure reported by analyst 1 is assigned both a TPM1 and an FNA1. The two structures reported by analyst 2 are assigned a TPM1 and a TPU1, respectively.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

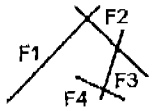
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

Fig. X2.6 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one structure and analyst 2 has reported four structures. Under the AHERA counting rules, analyst 1 is correct. The structure reported by analyst 1 is assigned a TPM1. The first structure reported by analyst 2 is labelled TPM1 and the remaining three reported structures are labelled FP1-FP3.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1			0	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1			1	Chr

a

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		FNA1	0	Chr

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

b

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		FP1	1	Chr

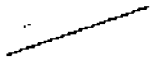
c

Fig. X2.7 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same asbestos-containing particle as indicated by the identification and orientation of the fiber and by the presence of a similar particle nearby. However, the dimensions reported by the analysts differ and analyst 1 has reported zero structures and analyst 2 has reported one structure. The verifying analyst should determine the correct length of the fiber and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 2 is assigned a TPU1 and analyst 1 an FNA1. c) A second possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 1 is assigned a TN1 and analyst 2 an FP1.

Analyst 1

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1			1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

a

Fig. X2.8 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Analyst 1 has reported one structure that analyst 2 has not reported. The verifying analyst should attempt to find the particle and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is assigned a TPU1 and analyst 2 is assigned an FNB1. c) Another possible outcome is that the reported structure is not located. Analyst 1 is assigned an NL. Other possibilities (not illustrated) are that analyst 1 is incorrect (the particle is then labelled FP) or that the structure is too contaminated for characterization (the particle is then labelled AMB).

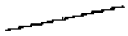
Analyst 1

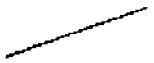
Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		TPU1	1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		FNB1 TPM2	1	Chr

b

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		NL1	1	Chr
1.0	0.1		TPM2	1	Chr

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

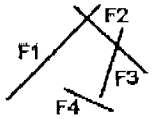
c

Fig. X2.8 (caption on previous page).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3			1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1		1	Chr
3	0.1	F2		1	Chr
2	0.1	F3		1	Chr
1	0.1	F4		1	Chr

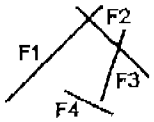
a

Fig. X2.9 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same particle as indicated by the identification and orientation of the fibers. However, analyst 1 has recorded all fibers as touching (or intersecting) and has therefore counted the fiber arrangement as one structure under the AHERA method. Analyst 2 has reported four structures. The verifying analyst should find and examine the arrangement in the TEM to determine if the fiber labelled as F4 by analyst 2 is touching or intersecting the fiber labelled as F3. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is then assigned a TPM1 and analyst 2 is assigned a TPM1 and three FPs. Other possibilities (not illustrated) are that analyst 2 is correct (the structures reported by analyst 2 are then assigned a TPM and 3 TPUs and the structure reported by analyst 1 is assigned a TPM) or that the particle is too contaminated for identification (the structure reported by analyst 1 is then assigned a TPM and those reported by analyst 2 are assigned a TPM and three AMBs).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

b

Fig. X2.9 (caption on previous page)

X3. SUMMARY OF THE PROCEDURE FOR COMPARISON OF TWO TEM ANALYSIS FORMS

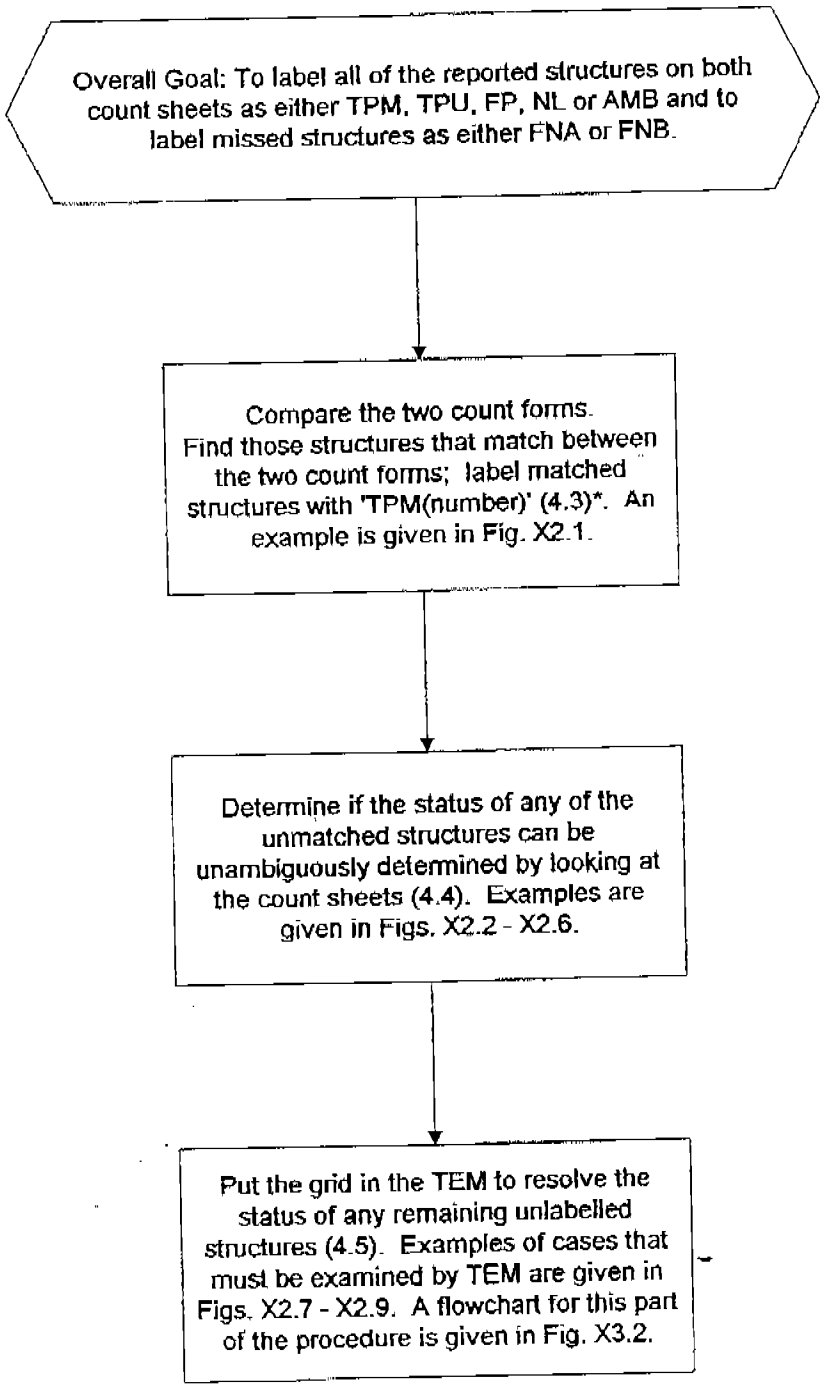


Fig. X3.1 Summary of the overall procedure for comparison of TEM analysis forms by the verifying analyst. *Numbers in parentheses in each block refer to the item number in the procedure.

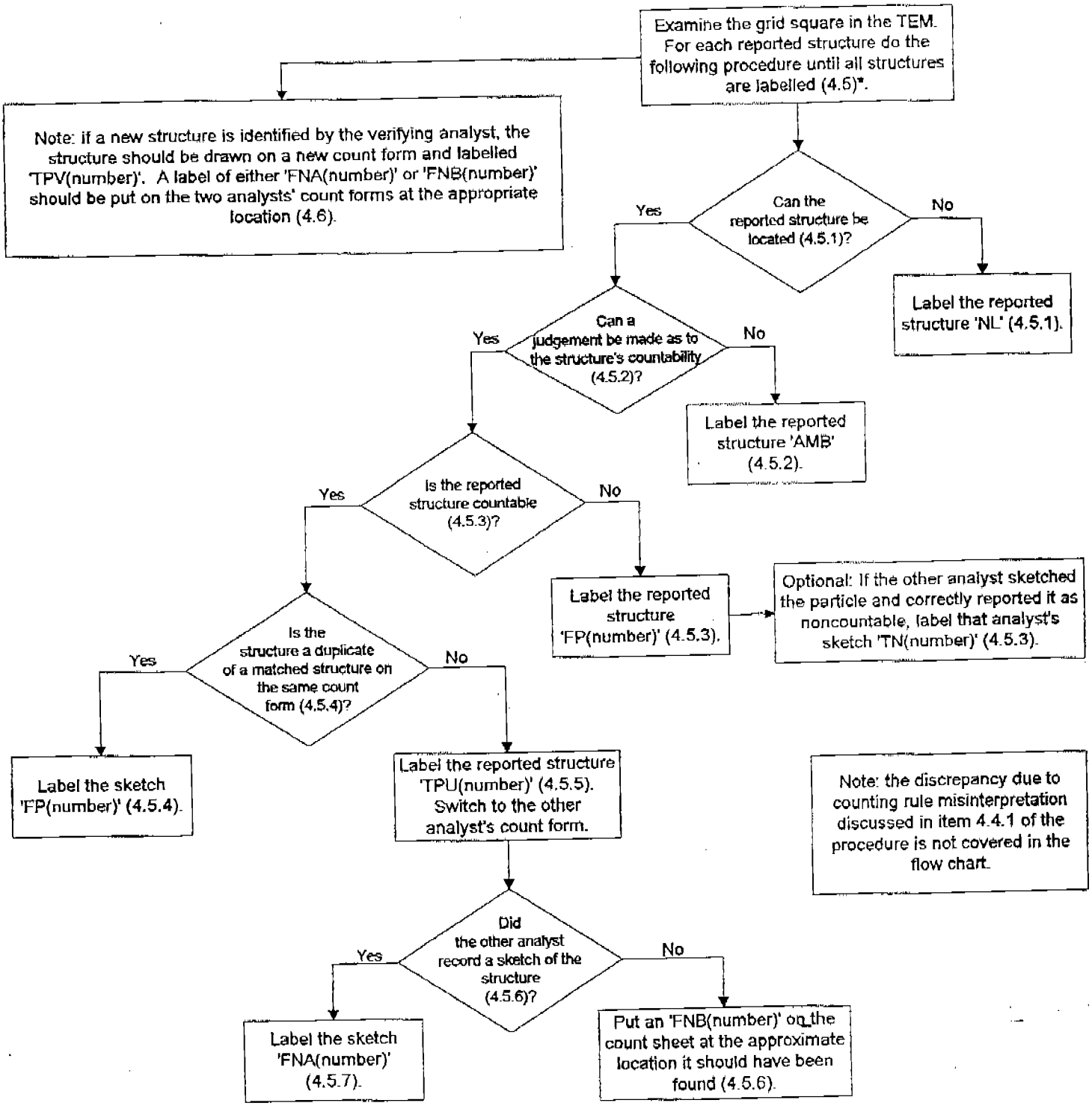


Fig. X3.2 Flowchart for examination of a structure in the TEM. The flowchart is an expansion of the last block in Fig. X3.1. *Numbers in parentheses in each block refer to the item number in the procedure.

ATTACHMENT 4

Statistical Comparison of Two Poisson Rates

1.0 INTRODUCTION

An important part of the Quality Control plan for this project is the repreparation and reanalysis of a number of TEM grids for quantification of asbestos fiber concentrations in air and dust. Because of random variation, it is not expected that results from repreparations samples should be identical. This attachment presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

2.0 STATISTICAL METHOD

This method is taken from "Applied Life Data Analysis" (Nelson 1982). Input values required for the test are as follows:

N1 = Fiber count in first evaluation
S1 = Sensitivity of first evaluation
N2 = Fiber count in second evaluation
S2 = Sensitivity of second evaluation

The test is based on the confidence interval around the ratio of the two observed Poisson rates:

Rate 1 = N1 · S1
Rate 2 = N2 · S2
Ratio = Rate 1 / Rate 2

$$Lower\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1}{N2+1}\right) / F\left[\frac{1+\gamma}{2}; 2\cdot N2+2, 2\cdot N1\right]$$

$$Upper\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1+1}{N2}\right)\cdot F\left[\frac{1+\gamma}{2}; 2\cdot N1+2, 2\cdot N2\right]$$

where γ is the confidence interval (e.g., 0.95) and F[δ; df1, df2] is the 100δth percentile of the F distribution with df1 degrees of freedom in the numerator and df2 degrees of freedom in the denominator.

If the lower bound of the ratio is > 1, then it concluded that rate 1 is greater than rate 2 at the 100(1-γ)% significance level. If the upper bound of the ratio is < 1, then it concluded that rate 1 is less than rate 2 at the 100(1-γ)% significance level. Otherwise, it is concluded that rate 1 and rate 2 are not different from each other at the 100(1-γ)% significance level.

Example:

N1 = 4 structures
S1 = 0.0001 $(cc)^{-1}$
Rate 1 = 4 · 0.0001 = 0.0004 s/cc

N2 = 6 structures
S2 = 0.001 $(cc)^{-1}$
Rate 2 = 6 · 0.001 = 0.006 s/cc

γ = 0.95

$$Lower\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4}{6+1}\right) / F\left[\frac{1+0.95}{2}; 2\cdot 6+2, 2\cdot 4\right] = 0.014$$

$$Upper\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4+1}{6}\right) \cdot F\left[\frac{1+0.95}{2}; 2\cdot 4+2, 2\cdot 6\right] = 0.281$$

In this example, because the upper bound of the ratio is < 1, it is concluded that Rate 1 (0.0004 s/cc) is less than Rate 2 (0.006 s/cc) at the 95% significance level.

3.0 REFERENCES

Nelson W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

ATTACHMENT 5

NVLAP Airborne Asbestos Proficiency Test 98-2:
Grid Orientation

Instructions for Form 1

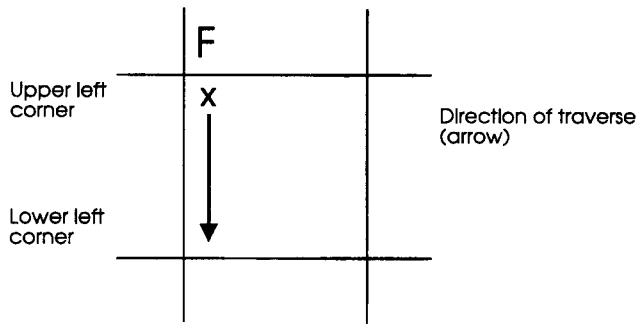
The following procedure is designed to ensure that all laboratories count the grid squares in the same orientation and scan direction to allow for verified analyses which will be performed in the next round of proficiency testing.

1. Put a grid into the TEM. Find a particle at the magnification typically used for asbestos analysis. Move the particle using one stage translation and record the direction of movement of the particle on *Form 1.* Move the particle using the other stage translation knob and record the direction of movement. Recording the two directions of movement should roughly form a cross. The cross represents the translation directions of your microscope at the magnification used for asbestos analysis. ***Draw the letter "F" onto the cross so the sides of the letter are parallel to the translation directions and the letter is upright and is not inverted.*** See the example on *Form 1.*

2. Decrease the magnification and locate the letter "F" on the finder grid. Increase the magnification of the TEM to that typically used for asbestos analysis by your lab, keeping the letter "F" in the field of view. Compare the orientation of the "F" to the cross drawn in step 1. If the letter "F" is not oriented as shown in your sketch, remove the specimen holder and rotate or invert the grid as necessary to correctly align the grid. This may require several iterations.

3. When the correct orientation is found, record the grid's position in the specimen holder as shown in the example of the second part of *Form 1.* Indicate in your drawing where the straight side and the notched portion of the grid are located. All grids analyzed in this proficiency test should be oriented in the same manner (always check that the letter "F" is in the correct orientation and that the X-Y translation directions allow translation roughly parallel to the grid bars).

4. The starting point of the traverse for structure counting must correspond to the upper left corner on the grid square. The "X" marks the starting corner of the traverse (your grid square may be at an angle to that shown in the example):



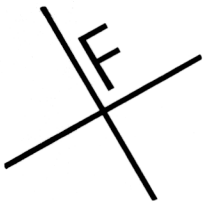
The initial direction of traverse must be from the upper left corner to the lower left corner of the grid square. If correctly oriented, the edge of the grid bar will remain in the field of view during the entire initial traverse (some allowance must be made for curvature or irregularly shaped grid bars.) If the grid is not oriented properly, go back to step 2.

NVLAP Lab Code: _____

Form 1. ***Grid Orientation***

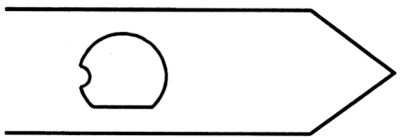
1. Sketch the orientation of the X-Y translation directions of the electron microscope as projected onto the electron microscope stage. Record the letter "F" as shown in the example below:

EXAMPLE:



2. Sketch below the orientation of the grid relative to the sample holder as shown in the example below:

EXAMPLE:



ATTACHMENT 6

Grid Opening Template for Sketching the Relative Position of Observed Structures

STRUCTURE LOCATIONS WITHIN GRID OPENING

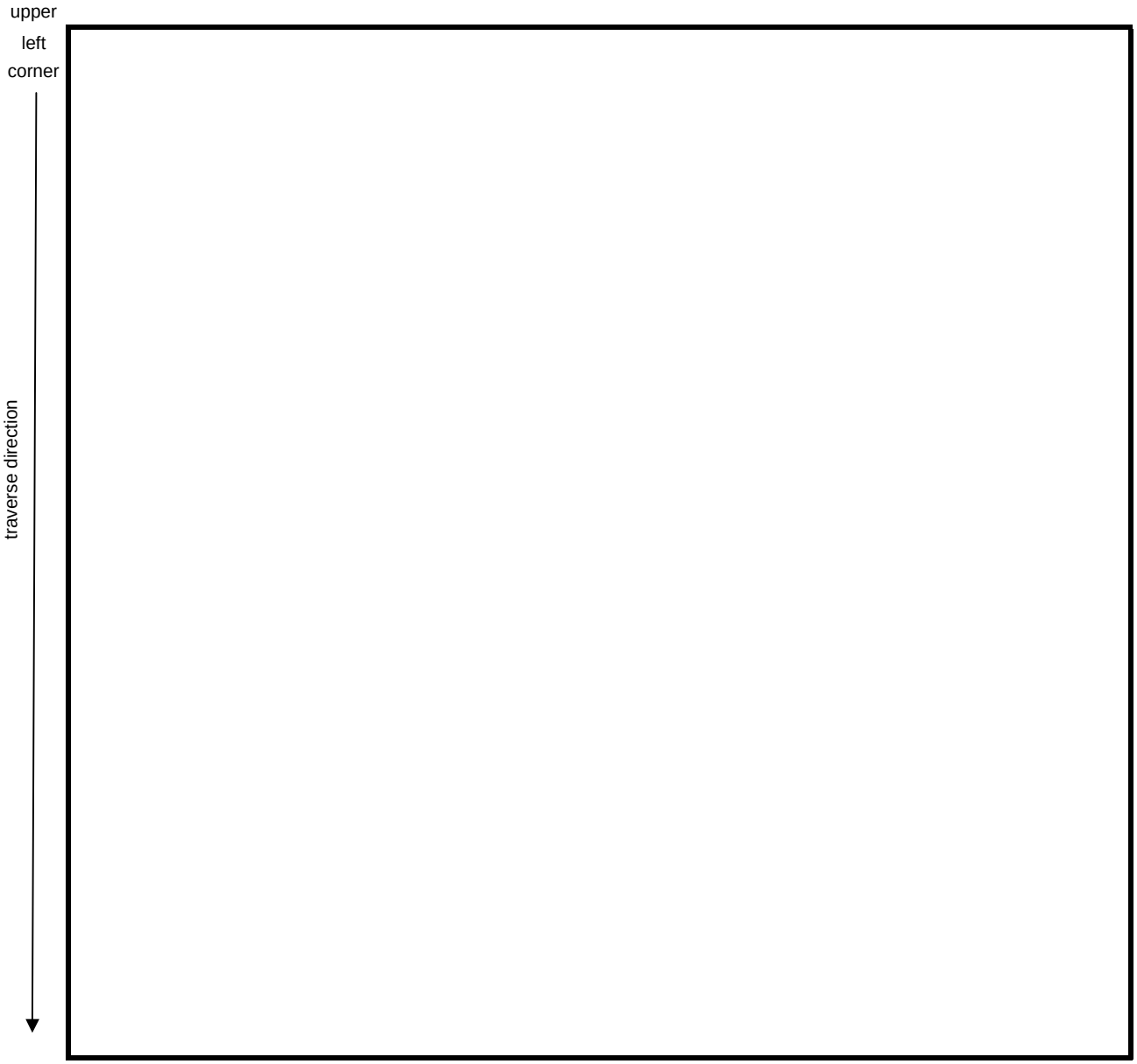
****NOTE: Sketches only need to be completed for interlab analyses and repreps associated with interlabs*

Lab Name: _____ Lab Job Number: _____

Index ID: _____ Lab Sample ID: _____

Lab QC Type (circle one): Reprep for interlab Interlab

Grid: _____ Grid Opening: _____



Comments:

Request for Modification
To
Laboratory Activities
LB-000030

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): [TEM-AHERA, TEM-ISO 10312], PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, [ASTM D5755-95], EPA/540/2-90/005a, Other: [EPA/600/R-94/134 (EPA 100.2)]

Requester: W.J. Brattin Title: Technical consultant
Company: Syracuse Research Corporation Date: 5 August 2003

Description of Modification:
All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance (morphology), and orientation relative to any nearby landmarks, if present.

Reason for Modification:
This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no loner need to be identified before analysis.

Potential Implications of this Modification:
There are no potential negative implications resulting from this standardization of QC procedures.

Laboratory Applicability (circle one): [All] Individual: _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

[Permanent] (complete Proposed Modification Section) Effective Date: 8/14/03 (insert based on date of final approval)
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: W J Brattin Date: 8/14/03
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 8/14/03
(Volpe: Project Technical Lead or designate)

Approved By: Mary Goldade Date: 8/14/03
(USEPA: Project Chemist or designate)

Autio, Anni

From: Goldade.Mary@epamail.epa.gov
Sent: Thursday, August 07, 2003 10:43 AM
To: Autio, Anni
Cc: Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo
Subject: EPA Comments: LB-000030 (Draft for review/comment)


LB-000030 v0 (MG 08-07-03).doc... pic08313.gif (3 KB)

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

One other point of clarification....when we discussed this, we were focused on AHERA. Just want to make sure it's OK w/ all to include TEM ISO on this list of circled methods. Thanks, Mary (See attached file: LB-000030 v0 (MG 08-07-03).doc) (Embedded image moved to file: pic08313.gif)

Request for Modification
To
Laboratory Activities
LB-000030

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): [TEM-AHERA, TEM-ISO 10312], PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, [ASTM D5755-95], EPA/540/2-90/005a, Other: [EPA/600/R-94/134 (EPA 100.2)]

Requester: W.J. Brattin Title: Technical consultant
Company: Syracuse Research Corporation Date: 5 August 2003

Description of Modification:
All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance, morphology and orientation relative to any nearby landmarks, if present.

Deleted: i

Reason for Modification:
This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no longer need to be identified before analysis and will be randomly selected by the laboratory's supervisor or designate following analysis.

Potential Implications of this Modification:
There are no potential negative implications resulting from this standardization of QC procedures, but a benefit is that samples selected for verified analyses will be unknown to the microscopist prior to analysis.

Laboratory Applicability (circle one): [All] Individual: _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

[Permanent] (complete Proposed Modification Section) Effective Date: (insert based on date of final approval)
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Deleted: _____

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

Autio, Anni

From: DeMalo, Robert [RDemalo@EMSL.com]
Sent: Thursday, August 07, 2003 11:20 AM
To: Goldade.Mary@epamail.epa.gov; Autio, Anni
Cc: Bob Shumate; LaCerra, Charles; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; EMSL Mobile Lab - Asbestos; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Richard Hatfield; Mahoney, Ron; Shu-Chun Su; Bill Longo
Subject: RE: EPA Comments: LB-000030 (Draft for review/comment)

I propose adding the word "morphology" as well into the description, as noted. I have no problem with including ISO to this procedure.

-----Original Message-----
From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]
Sent: Thursday, August 07, 2003 10:43 AM
To: Autio, Anni
Cc: Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo
Subject: EPA Comments: LB-000030 (Draft for review/comment)

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

One other point of clarification....when we discussed this, we were focused on AHERA. Just want to make sure it's OK w/ all to include TEM ISO on this list of circled methods. Thanks, Mary (See attached file: LB-000030 v0 (MG 08-07-03).doc) (Embedded image moved to file: pic08313.gif)

Autio, Anni

From: Raney, Mark [RANEY@VOLPE.DOT.GOV]
Sent: Thursday, August 14, 2003 10:41 AM
To: 'Goldade.Mary@epamail.epa.gov'; Autio, Anni
Cc: Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Raney, Mark; Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo
Subject: RE: EPA Comments: LB-000030 (Draft for review/comment)

LB-000030 v0 (MR 08-14-03).doc...

I concur with Mary's recommendations and mark-ups. The attached version also includes Rob Demalo's recommendation of adding morphology under the description section. Bill please finalize, sign and send it through the signature process. To expedite the process could you get Mary to sign before providing the original on for my signature. Let me know if you have any questions.

Thanks,

Mark.

-----Original Message-----
From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]
Sent: Thursday, August 07, 2003 10:43 AM
To: Autio, Anni
Cc: Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo
Subject: EPA Comments: LB-000030 (Draft for review/comment)

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

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Request for Modification
To
Laboratory Activities
LB-000030

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): [TEM-AHERA, TEM-ISO 10312], PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, [ASTM D5755-95], EPA/540/2-90/005a, Other: [EPA/600/R-94/134 (EPA 100.2)]

Requester: W.J. Brattin Title: Technical consultant
Company: Syracuse Research Corporation Date: 5 August 2003

Description of Modification:
All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance, morphology and orientation relative to any nearby landmarks, if present.

Deleted: i

Reason for Modification:
This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no longer need to be identified before analysis and will be randomly selected by the laboratory's supervisor or designate following analysis.

Potential Implications of this Modification:
There are no potential negative implications resulting from this standardization of QC procedures, but a benefit is that samples selected for verified analyses will be unknown to the microscopist prior to analysis.

Laboratory Applicability (circle one): [All] Individual: _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

[Permanent] (complete Proposed Modification Section) Effective Date: (insert based on date of final approval)
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Deleted: _____

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

Request for Modification
to
Laboratory Activities
LB-000066c

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other:_____

Requester: W. Brattin Title: Technical Consultant
Company: Syracuse Research Corporation Date: 09/11/2007

Description of Modification:
This temporary modification applies to all investigative samples (as defined by the most recent version of LB-000053) evaluated at the Libby Superfund site. Based on this temporary modification, all analytical laboratories shall: 1) begin to utilize the structure comment field to further characterize particles with regard to the levels (presence/absence) of the sodium and potassium peaks observed in the EDS spectrum; 2) record on the data sheets all NAM particles that are "close calls" (defined in attachment 1); 3) increase the frequency that EDS spectra are saved for "LA" and "close call" structures; 4) increase the frequency that photographic images of particle morphology are recorded for "LA" and "close call" structures, and 5) utilize the comment field to record mineral type of each recorded particle, including LA, OA, C and "close call" NAM particles.

Reason for Modification:
Studies of asbestos from the mine in Libby indicate that the asbestos spans several different mineralogical classes, including winchite and richterite (these are the primary forms) as well as tremolite and possibly actinolite (these are minor forms) (Meeker et al, 2003). Consequently, all analytical laboratories supporting the Libby project are currently directed to classify as "LA" any particle in an investigative sample that a) meets morphological requirements (e.g., length ≥ 0.5 um, aspect ratio ≥ 3:1), b) has an SAED diffraction pattern that is consistent with amphibole, and c) has an EDS spectrum that is consistent with the range of mineral forms observed in the mine in Libby (USEPA 2005). To date, this method for designating "LA" to a particle has worked well for samples collected at the Libby Site. However, a recent project that included collection of air samples from locations outside of Libby highlighted a potential limitation of this approach. That is, tremolite and actinolite are included in the "LA" suite and are found in Libby, but these types of fibers may also occur as the result of releases from sources that are not related to the mine in Libby (e.g., commercial products or natural sources). Also, some other minerals (e.g., pyroxenes) are sometimes difficult to distinguish from actinolite and tremolite (Bern et al. 2002). Because mineralogical data may or may not inform our understanding of the toxicity of LA, delineating amongst these mineral types is desirable at this stage of data collection. Therefore, the primary focus of this temporary modification is to collect more detailed data on the frequency of occurrence of sodium and potassium-containing particles both for samples from Libby and for samples from other locations.

Potential Implications of this Modification:
This temporary modification does not change any current procedures other than to require more detailed recording of data on particles observed under TEM. These additional requirements are not associated with a significant increase in time or cost of analysis. Hence, there are no negative implications of the modification.

Laboratory Applicability (circle one): [All] Individual(s) _____

Duration of Modification (circle one):

[Temporary] Date(s): 09/12/2007 until notified

Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: _____

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

[Not Applicable] **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

See Attachment 1

Note: This modification (LB-000066c) **supersedes** LB-000066b.

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 9/12/07
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: 9/11/07
(USEPA: Project Chemist or designate)

REFERENCES

Bern A, Meeker G, Brownfield I. 2002. Guide to Analysis of Soil samples from Libby, Montana for Asbestos Content by Scanning Electron Microscope and Energy Dispersive Spectroscopy. U. S. Geological Survey Administrative Report. October 17, 2002.

Meeker GP, Bern AM, Brownfield IK, Lowers HA, Sutley SJ, Hoeffen TM, and Vance JS. 2003. The Composition and Morphology of Amphiboles from the Rainy Creek Complex, Near Libby Montana. American Mineralogist 88:1955-1969.

USEPA. 2005. EDS Spectra Characteristic Study for Libby-Type Amphiboles. Report prepared by Syracuse Research Corporation, Denver CO, for USEPA, Region 8, Denver CO. March 15, 2005.

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

ATTACHMENT 1

1. Continue to classify structures as LA, OA, or C in accord with current procedures.

2. For all NAM particles that were "close calls" (i.e., they required careful assessment to determine they were not LA or OA), record the NAM particle on the bench sheet. Be sure to place a zero in the "total" column to ensure the particle is not counted as an asbestos fiber. NAM particles such as vermiculite, biotite, hydrobiotite, gypsum, titanium and other minerals that are clearly not amphibole should not be recorded.

3. For all particles that are recorded (including NAMs), use the structure comment field to record one of the following comments:

Code	Meaning
NaK	Na and K are both clearly present
NaX	Only Na is clearly present
XK	Only K is clearly present
XX	Na and K are not clearly present

4. For all particles that are recorded, whenever possible, use the structure comment field to identify a probable mineral classification. Use the designation "WRTA" (winchite/richterite/tremolite/actinolite) to indicate a particle that is consistent in morphology and chemical composition with a particle that is likely to have originated from the vermiculite mine in Libby. This will include most NaK particles and may include some NaX and some XK particles. It is unlikely that this will include any XX particles. For all other particles, use the following codes:

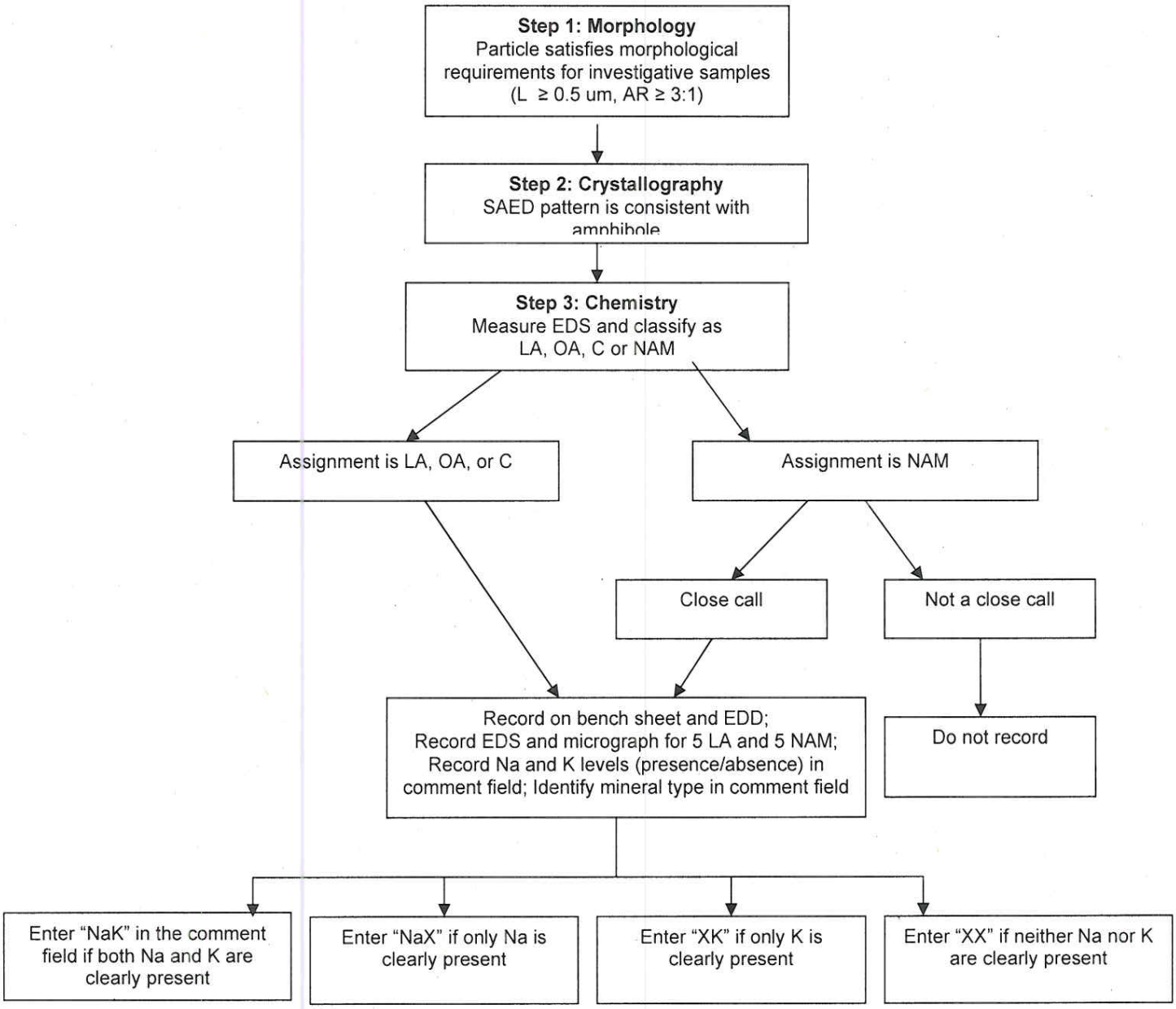
AC – actinolite
TR – tremolite
AT – actinolite/tremolite (too close to call)
AM – amosite
AN – anthophyllite
CR – crocidolite
PY – pyroxene
UN – Unknown

5. Increase the frequency that EDS spectra are recorded (saved). For each sample, record the EDS for each LA and each "close call" particle, up to a maximum of 5 LA and 5 "close call" particles per sample. To the extent practical, collect the EDS spectrum for a sufficient length of time that key peaks (e.g., sodium, potassium, aluminum), if present, can be clearly distinguished from background. Be sure that each EDS spectrum that is recorded can be linked to a specific particle in the EDD.

6. Increase the frequency that photomicrographic images of particle morphology are collected. For each particle for which an EDS spectrum is collected (up to 5 LA and 5 "close call" NAM, as discussed above), also record a photomicrograph of the same structures. Use the structure-specific comment field to record the photo identification number of each structure that is photographed. Convert all photographs to high quality electronic images (e.g., by scanning), and transmit the photos to CDM for evaluation.

7. Figure 1 provides a flow chart that summarizes the process implemented by this temporary modification.

FIGURE 1
FLOW CHART SUMMARIZING THIS TEMPORARY MODIFICATION



Request for Modification
to
Laboratory Activities
LB-000085

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): [TEM-AHERA] [TEM-ISO 10312] PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 [ASTM D5755] [EPA/540/2-90/005a] SRC-LIBBY-03
Other: All TEM and SEM Methods supporting Libby site investigative or Libby Action Plan (LAP) sample analysis

Requester: Mary Goldade Title: Senior Environmental Scientist/Chemist
Company: Environmental Protection Agency, Region 8 Date: April 2, 2008

Description of Modification:
Laboratories conducting transmission electron microscopy (TEM) or scanning electron microscopy (SEM) analysis in support of either the Libby Site (all operable units, including Troy) or Libby Action Plan shall perform analysis of a reference standard to calibrate the energy dispersive x-ray spectrometry (EDS) analysis. The reference standard, a glass material referred as BIR-1G, was created by the USGS. It is recommended for use for Libby Amphibole analysis because it contains sodium (Na) and potassium (K) at known levels. Na and K are important elements used in Libby Amphibole identification by EDS. The BIR-1G standard was freezer-milled by EMSL to create particles for EM analysis. While generation of thin sections of the BIR-1G using a microtome was not feasible due to the expense, analysis of the BIR-1G in particulate form is useful in standardizing the elemental measurements of the EDS and understanding the inherent variability in the EDS measurements.

The BIR-1G shall be tested daily (on days that the TEM scope is used for analysis of Libby samples) and must meet acceptance criteria prior to analysis of any field samples. Laboratories shall record the calibration information in accord with Attachment 1. As seen, not only does Attachment 1 provides the details for populating the electronic disk deliverable (EDD) used in recording the calibration information, but Attachment 1 also describes the process for generating acceptance criteria for the BIR-1G standard for each individual instrument.

Reason for Modification:
The modification provides for a standardized process for performing and recording calibration standards for EDS during Libby Amphibole analysis.

Potential Implications of this Modification: There are no negative implications to this modification. Positive impacts include a standardized process for: (1) daily calibration of a standard for the EDS used in Libby Amphibole identification; (2) reporting results of BIR-1G measurements; and (3) generating acceptance criteria for the BIR-1G standard over time.

Laboratory Applicability (circle one): [All] Individual(s) _____

This laboratory modification is (circle one): **[NEW]** **APPENDS to** _____ **SUPERCEDES** _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

[Permanent] (Complete Proposed Modification Section) Effective Date: April 30, 2008
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _____N/A_____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: _____
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

ATTACHMENT 1

Analyzing the BIR-1G Standard

- The BIR-1G standard shall be tested daily (on days that either the SEM or TEM microscope is used for analysis of Libby samples), prior to analyzing any field samples. Analyze for the compounds Na_2O, MgO, Al_2O_3, SiO_2, K_2O, CaO, TiO_2, MnO, and FeO. It is suggested that the reference publication for BIR-1G be reviewed. It is available in Volume 2 of the Analytical Guidance Documents, Tab 35, provided by CDM.
- Set up TEM instrument and orient for typical Libby field samples.
- Record the TEM instrument details in the BIR-1G Electronic Data Deliverable (EDD) spreadsheet (see most recent version of Excel file "BIR-1G EDD.xls"). Note: Use one spreadsheet per TEM instrument.
- For each daily BIR-1G evaluation, select one particle and record the measured weight % for each compound ***as oxide weight %*** in the BIR-1G EDD. Note: When recording oxide weight %, enter results as a percentage not fractions (i.e., for 30%, enter 30 not 0.3).
- When selecting particles for analysis:
    - Choose particles in the middle of the grid opening and in the center of the grid.
    - Particles should not be in close proximity to the grid bar or neighboring particles.
    - Randomly select particles within different grid openings for each analysis.
- For selected particles, focus the beam on the thin edge, not the center of the particle.
- Continue analysis until a maximum peak height count of at least 1,000 is achieved for silicon (Si). This total Si count should be sufficient to achieve optimum instrument testing conditions. It is recognized that this total Si count may not be equivalent to typical analytical conditions for field samples.
- On a monthly basis, the EDD for each TEM instrument should be provided to EPA (or designated contractors).

Acceptance Criteria

- Acceptance criteria will be TEM instrument- and element-specific and will be derived from measured results.
    - Results that are within ± 1 standard deviation of the nominal will be ranked as acceptable.
    - Results that are outside ± 1 standard deviation but within ± 2 standard deviations of the nominal will be ranked as within the warning level.
    - Results that are outside ± 2 standard deviations of the nominal will be ranked as a failure.
- The potential bias of measured results will be assessed based on a frequency evaluation of results above and below the nominal.
- As needed, EPA will re-evaluate and revise the acceptance criteria to optimize program goals.

Corrective Action

In the event that analysis results of the BIR-1G fall outside of the acceptance criteria, there should be a structured, progressive response. First, confirm that the detector/x-ray system has satisfied the acceptance criteria in the past. Next, confirm that the settings for the x-ray analysis software are correct (e.g. bias, scale). Finally, de-ice the LN2 dewar (unless it is a dry system) and carefully clean the window.

If these actions fail to rectify the problem, it will probably be necessary to send the detector/x-ray out to be serviced. The actions taken by the servicing company may include such things as baking the detector, renewing the vacuum in the dewar, checking the pre-amp or actual x-ray system for hardware defects, or replacing the crystal and/or FET (field effect transistor). In most instances the fault will not lie in the window unless the integrity of the window is compromised.

Upon the return and re-installation of the detector, re-run the BIR-1G standard to confirm that corrective action measures have resolved analysis issues.